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EDGEWOOD ARSENAL  
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## COMPOUNDING TECHNIQUES FOR ABSORBENT DECONTAMINANTS

Final Report  
(August 1970 - November 1971)

by  
Dewey P. Parks

January 1972



DEPARTMENT OF THE ARMY  
EDGEWOOD ARSENAL  
Development and Engineering Directorate  
Edgewood Arsenal, Maryland 21010

Contract Number DAAA15-70-C-0362

Brunswick Corporation  
Technical Products Division  
Marion, Virginia  
24354

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EDGEWOOD ARSENAL  
CONTRACT REPORT

(18) EACR-1318-3

(19)

(14) BR-303

(2) COMPOUNDING TECHNIQUES FOR  
ABSORBENT DECONTAMINANTS.

(1) Final Report.

(August 1970 - November 1971)

by

(12) Dewey P. Parks and Robert L. Copeland

(11) Jan 1972

DEPARTMENT OF THE ARMY

EDGEWOOD ARSENAL

Development and Engineering Directorate

Edgewood Arsenal, Maryland 21010

(15) Contract Number DAAA15-70-C-0362

Project No. 233-DP-0 (REF 0362)

(16) DA-

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## FOREWORD

The work described in this report was authorized under Contract DAAA15-70-C-0362, Compounding Techniques for Absorbent Decontaminants. This work was started in August 1970 and completed in November 1971.

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## ACKNOWLEDGMENTS

Acknowledgment for technical assistance to Mr. Harold Cook, Dr. Charles Greene, and Mr. Robert Hurley.

## DIGEST

The objective of the program was to develop decontaminating systems for persistent chemical agents using micronized carbon. These systems must be effective in absorbing chemical agents and designed to the following goals.

- Act rapidly over a wide temperature range
- Be non-toxic and remain stable
- Not damage material and equipment on contact
- Be easily and rapidly applied to vehicular equipment, clothing and ancillary equipment.

The method of study was to select key ingredients, i.e., activated carbons, thixotropic agents, dispersants, pigments and solvent/antifreeze and formulate a system containing a minimum of 25% active carbon. It should be easily applied by spraying to a coverage of 4 mg/cm<sup>2</sup> and remain stable (not settle during static storage of at least two years). Key methods of investigation included evaluation of mixing techniques, effects of various thixotropes and dispersant on viscosities and stability and the evaluation of spray equipment for optimization of techniques as well as equipment. In addition, studies were conducted whereby dry formulation of the same key ingredients were formulated using high shear dry mixers. These compounds were tested for uniformity, adhesion to woven surfaces and effective coverages in the 4 mg/cm<sup>2</sup> range.

The results of these studies show that formulations containing approximately 35% active carbon can be easily sprayed to the desired coverage and will remain stable for the desired two year period. All of the program goals outlined above were met or exceeded by this study. Results also show dry formulations can be prepared by a special screened milling process to give the desired uniformity, color and adhesion.

It is concluded that in selecting the optimum ingredients to prepare a stable, sprayable thixotrope that will meet all of the Edgewood requirements, the following materials have shown to be superior: coal, petroleum, coconut and wood based activated carbons are suitable as agent absorbents; however, the coal and petroleum-based appear slightly superior. Thixotropes appear to be limited to the inorganic type with bentonite showing superior thixotropic properties. Dispersants are effective in lowering viscosities with the sodium salts of polymerized substituted benzoid alkyl sulfonic acid being most effective. Color can be obtained with iron oxide and 1/2% sodium chromate or sodium silicate to inhibit rust. A homogenizer mixer will produce uniform mixtures of the wet-system and a special screen milling machine will prepare satisfactory dry formulations.

# TABLE OF CONTENTS

<u>Section</u>	<u>Description</u>	<u>Page</u>
I	INTRODUCTION AND BACKGROUND .....	9
II	PHASE A - VEHICULAR DECONTAMINATION .....	9
	A. Materials .....	9
	B. Mixing and Testing Equipment .....	12
	C. Formulation and Experimentation .....	15
	1. Organic Polymer Thixotropes .....	15
	2. Inorganic Thixotropes .....	19
	a. General Outline of Investigation .....	19
	b. Selection of Mixing Equipment .....	23
	c. Evaluation of Each Based Carbon .....	25
	d. Formation of Thixotropic Gels .....	74
	D. Spraying and Adhesion Testing .....	78
	1. Spray Experiments .....	78
	2. Adhesion Tests .....	81
III	PHASE B - CLOTHING AND INDIVIDUAL EQUIPMENT DECONTAMINATION .....	87
	A. Basic Materials, Equipment and Methods of Investigation .....	87
	1. Government Supplied Materials .....	87
	2. Inert Filler Materials .....	87
	3. Mixing Equipment .....	88
	4. Auxiliary Equipment .....	88
	B. Formulation and Testing of Dry Carbon Mixtures .....	89
	1. Microscopic Examination .....	89
	2. Preparation of Wet and Dry Formulation .....	93
	3. Abrasion Tests .....	94
	4. Other Fibrous Materials .....	101
	C. Carbon Flow Control From Applicator Bag .....	102
IV	CONCLUSIONS AND RECOMMENDATIONS .....	102
V	DISTRIBUTION LIST .....	105
VI	DOCUMENT CONTROL DATA, FORM 1473 .....	107

## LIST OF TABLES

<u>Table</u>	<u>Description</u>	<u>Page</u>
I	Static Storage Tests of Carbon/Carbopol 934 Thixotropes .....	20
II	Static Storage Tests of Carbon/Carbopol 941 Thixotropes .....	21
III	Preparation of Carbon Suspensions .....	24
IV	Carbon Suspensions Prepared with the L-1 Homogenizer .....	26
V	Carbon Suspensions Prepared with a Cowles Dissolver .....	27
VI	Carbon Suspensions Prepared with a Ball Mill .....	28
VII	CWS Carbon-Thixotrope Evaluation .....	32
VIII	CWS Carbon-Dispersant Evaluation .....	33

# LIST OF TABLES (CONT'D)

<u>Table</u>	<u>Description</u>	<u>Page</u>
IX	CWS Carbon-Dispersant Evaluation .....	34
X	CWS Carbon-Percent Dispersant Evaluation .....	36
XI	CWS Carbon-Corrosion Inhibitor .....	37
XII	CWS Carbon-Mixing Time Study .....	38
XIII	CWS Carbon-Pigment Evaluation .....	39
XIV	Darco KB Carbon-Thixotrope Evaluation .....	43
XV	Darco KB Carbon-Dispersant Evaluation .....	44
XVI	Darco KB Carbon-Percent Dispersant .....	45
XVII	Darco KB Carbon-Percent Dispersant .....	46
XVIII	Darco KB Carbon-Corrosion Inhibitor .....	49
XIX	Darco KB Carbon-Mixing Time Evaluation .....	50
XX	Darco KB Carbon-Pigment Evaluation .....	51
XXI	ACC Carbon-Thixotrope Evaluation .....	55
XXII	ACC Carbon-Dispersant Evaluation .....	56
XXIII	ACC Carbon-Dispersant Evaluation .....	57
XXIV	ACC Carbon-Percent Dispersant Evaluation .....	58
XXV	ACC Carbon-Corrosion Inhibitor Evaluation .....	60
XXVI	ACC Carbon-Mixing Time Evaluation .....	61
XXVII	ACC Carbon-Corrosion Inhibitor Evaluation .....	62
XXVIII	UU Carbon-Thixotrope Evaluation .....	66
XXIX	UU Carbon-Dispersant Evaluation .....	67
XXX	UU Carbon-Dispersant Evaluation .....	68
XXXI	UU Carbon-Percent Dispersant Evaluation .....	69
XXXII	UU Carbon-Corrosion Inhibitor Evaluation .....	71
XXXIII	UU Carbon-Mixing Time Study .....	72
XXXIV	UU Carbon-Pigment Evaluation .....	73
XXXV	Formation of Thixotropic Gel .....	75
XXXVI	Static Storage Test of Carbon Thixotropes .....	77
XXXVII	Abrasion Resistance of Activated Carbon Deposited on Lacquer Panels .....	86
XXXVIII	Percent Carbon Retention vs. RPM of Test Apparatus .....	96
XXXIX	Percent Carbon Retention of Wet and Dry Prepared Formulations .....	97
XL	Effect of Moisture Content on Percent Retention to Woven Fabric .....	98
XLI	Effect of Pulverizing Screen Size on Percent Retention to Woven Fabrics .....	99
XLII	Percent Retention of Carbon/Asbestos to Different Types of Woven Uniform Material .....	101



## LIST OF FIGURES

<u>Figure</u>	<u>Description</u>	<u>Page</u>
1	L-1 Homogenizer Mixer .....	13
2	Viscosity Vs. Shear Rate in Measuring the Degree of Thixotropy .....	22
3	Effect of % Dispersant on CWS Carbon Slurries .....	35
4	Effect of % Dispersant on Darco KB Carbon Slurries .....	48
5	Effect of % Dispersant on ACC Carbon Slurries .....	59
6	Effect of % Dispersant on UU Carbon Slurries .....	70
7	Spray Testing Apparatus .....	79
8	Carbon Coated Panel with Test Tabs .....	79
9	Close-Up of Activated Carbon Coating Sprayed with Standard Sprayer Apparatus .....	83
10	Close-Up of Activated Carbon Coating Sprayed with DeVilbiss MBC Spray Gun .....	83
11	Abrasion Test Panels Prior to Spraying with Activated Carbon .....	84
12	Abrasion Test Panels with 3-4 mg/cm <sup>2</sup> Coating of Activated Carbon .....	84
13	Abrasion Test Set-up Using Instron Test Machine .....	85
14	Close-Up Showing 100 Gram Weight and Terry Cloth .....	85
15	Crysotile (Type 4A3) Asbestos as Received from Vendor .....	90
16	4A3 Asbestos Fibers After Milling Through a .010" x .500" Slotted Screen .....	90
17	4A3 Asbestos Fibers After Milling Through a .032" Perforated Screen .....	91
18	4A3 Asbestos Fibers After Milling Through a .040" Perforated Screen .....	91
19	4A3 Asbestos Fibers After Milling Through a .062" Perforated Screen .....	92
20	Amphibole Type Asbestos (Shorts) as Received From the Supplier .....	92
21	Abrasion Test Apparatus .....	95
22	Foam Covered Test Cylinder with Fabric Samples .....	95
23	Quarzel-Treated Nyco Sateen as Received .....	100
24	Nyco Sateen Impregnated with 4 mg/cm <sup>2</sup> of Carbon/Asbestos Mixture .....	100
25	Nyco Sateen After Abrasion Test .....	100

## COMPOUNDING TECHNIQUES FOR ABSORBENT DECONTAMINANTS

### I. INTRODUCTION

The objective of this program is to develop a universal decontaminating system for persistent chemical agents using micronized carbon. The decontaminating system must be effective in absorbing the chemical agents and is designed to the following goals:

- Act rapidly over a wide temperature range
- Be non-toxic
- Not damage material and equipment on contact
- Be easily and rapidly applied to vehicular equipment, clothing and ancillary equipment.

This study was particularly directed toward the application of activated carbon to vehicular equipment by spraying and to clothing and small equipment by dusting or rubbing. The program was conducted in three phases; i.e., (A) Vehicular decontaminants in the form of a sprayable suspension, (B) Clothing and individual equipment decontaminants in the form of dry powders, and (C) Scale-up methods and equipment for large volume manufacture and dispensing.

At the direction of Edgewood Arsenal, priority was placed on the successful completion of Phases A and B and if time and money permitted, Phase C was to be investigated.

The work performed during this contract shows how the key ingredients - activated carbon, thixotropic agents, dispersants, pigments, and solvent/antifreeze - are combined into a system to accomplish the objectives outlined above. Numerous experiments, which resulted in varying degrees of success at accomplishing the combined systems goal, are discussed in this final report.

### II. PHASE A - VEHICULAR DECONTAMINATION

This decontaminant was formulated from active carbon, a dispersant, pigment to give an olive green color, a thixotrope, a corrosion inhibitor, alcohol for antifreeze and water. The system must contain a minimum of 25% active carbon, be easily applied by spraying to give a coating of 4 mg/cm<sup>2</sup>, and remain stable (not settle) during static storage for at least two years.

#### A. Materials

To begin the investigation, candidate materials were selected for initial investigation. Based on previous research completed by Edgewood Arsenal and previous work by Brunswick Corporation, the following candidate materials were selected.

Activated Carbons - Carbons derived from wood, coal, petroleum coke and coconut shell were investigated. These particular carbons were selected on the basis of their gas vapor absorptive properties which are of utmost importance in this application. Previous research has shown the optimum particle size to be in the 1-2 $\mu$  range with any remaining particles being less than 20 $\mu$ . The carbons evaluated in this program were as follows:

<u>Carbon</u>	<u>Particle Size as Supplied</u>	<u>CCL<sub>4</sub> Activity</u>	<u>pH</u>	<u>Surface Area, Sq. Meters/g.</u>
<u>Coal Base</u>				
CWS* (Pittsburgh Carbon Co.)	1-2 $\mu$	65%	10.0	1000-1100
<u>Coconut Shell Base</u>				
Type UU (Barnebey-Cheney)	50 x 200	100%	10.4	1400-1600
<u>Petroleum Coke</u>				
Type ACC (Union Carbide)	Granular	63%	10.4	980-1080
<u>Wood Base</u>				
DARCO KB (Atlas Chemical Co.)	1-2 $\mu$	84%	5.2	600-700
*Supplied by Edgewood Arsenal				

Prior to formulating any carbon suspensions, particle sizes of the petroleum coke and coconut shell based were reduced to the desired 1-2 $\mu$  range. This was done by pulverizing with a hammer mill to reduce the particle size to approximately 40 $\mu$ . The additional reduction was completed by ball milling for 4-1/2 hours. Particle sizes were then measured by a Sub-Sieve Sizer. The average particle size of each of the two sized carbons was in the 1-2 $\mu$  range.

Thixotropic Agents - By incorporating a thixotropic agent into the system, the viscosity of the carbon suspensions can be adjusted high enough to prevent settling of the carbon particles during static storage, yet remain fluid enough for spray application when shear is applied. Based on previous Brunswick development work with thixotropic agents in liquid propellants, plus earlier research by Edgewood Arsenal, the following candidate thickening agents were selected for study.

<u>Thixotropic Agent</u>	<u>Type/Manufacturer</u>
Carboxy Vinyl Polymer System (water soluble)	Organic, Carbopol R/ B. F. Goodrich Co.

<u>Thixotropic Agent</u>	<u>Type/Manufacturer</u>
Microcrystalline Silica	Inorganic, Avibest C/ FMC Corp.
Micromesh Montmorillonite	Inorganic, Bentonite/ Van Horn Metz Co.
Resin Grade Asbestos	Inorganic, Calidria RG-244/Union Carbide Co.
Pyrogenic (Fumed) Silica	Inorganic, Aerosil COK-84/ DeGussa, Inc.
Microcrystalline Nylon	Organic, Aviamide 6-P Nylon/ FMC Corp.

Dispersants - To aid in obtaining low viscosities and to permit maximum percent carbon loading, dispersing agents were evaluated. With the majority of the carbons under investigation being basic, (pH ~ 10) solid, anionic dispersants were used to effectively separate each of the carbon particles which increased the stability of the mixture. Those investigated were:

<u>Dispersant</u>	<u>Trade Name/Manufacturer</u>
Polymerized Salt of Alkyl Naphthalene Sulfonic Acid	DAXAD 11 - W. R. Grace Co.
Partially Desulfonated Sodium Liquosulfonate	Marasperse CB - American Can Company
Sodium Salts of Polymerized Substituted Benzoid Alkyl Sulfonic Acid	DARVAN No. 2 - R. T. Vanderbilt Company
Water Miscible Polymer	Nopcosant K - Nopco Chemical Company

The effects of adding each of the dispersing agents to the carbon suspensions are discussed in the experimentation section.

Pigments - To produce an olive green color, pigments were selected and evaluated on the basis of "coloring power", i.e., the pigment which at the lowest weight percentage gave an olive green color.

<u>Pigment</u>	<u>Trade Name/Manufacturer</u>
Iron Oxide	Mapico 1100 Yellow/Columbian Carbon Company
Phthalocyanine Green	Color No. W-6012/Harshaw Chemical Company

<u>Pigment</u>	<u>Trade Name/Manufacturer</u>
Cadmium Yellow	Color No. 1489/Harshaw Chemical Company
Cadmium Yellow	Color No. AZO W-1215/Harshaw Chemical Company
Cadmium Orange	Color No. 1510/Harshaw Chemical Company
Cadmium Sulfide	Cadmium Yellow/Harshaw Chemical Company

Corrosion Inhibitor - To inhibit rust and corrosion following application of the decontaminant to a surface, the following materials were selected and evaluated.

<u>Type</u>	<u>Source</u>
Sodium Chromate ( $\text{Na}_2\text{CrO}_4$ )	Fisher Scientific
Sodium Silicate ( $\text{Na}_2\text{SiO}_3$ )	Fisher Scientific

Solvent-Antifreeze - As a carrier and antifreeze, the following was used.

Water - Demineralized	60 parts by volume
Methyl Alcohol	40 parts by volume

#### B. Mixing and Testing Equipment

Different pieces of equipment were selected and tested for efficiency and ease of mixing. Each piece of equipment was tested by mixing a formulation for given time intervals, then measuring the viscosity, followed by visual inspection and uniformity. The equipment tested is as follows:

Ball Mill - Formulations were ball milled a minimum of 48 hours using a 2 liter jar with approximately 1 pint of 1/2" diameter balls. The viscosity and stability data obtained from these experiments were used for comparison both to work done by Edgewood Arsenal and to the mixing efficiency of the other types of equipment.

Homogenizer Mixer - Figure I shows the Gifford-Wood, Model L-1 Homogenizer. It is characterized by a fixed clearance between a turbine and a stator head. The skewed turbine develops a pressure differential between the bottom of the turbine and the surface of the slurry. Unmixed material is continuously drawn from the bottom and forced through restricted openings in the stator striking the deflector

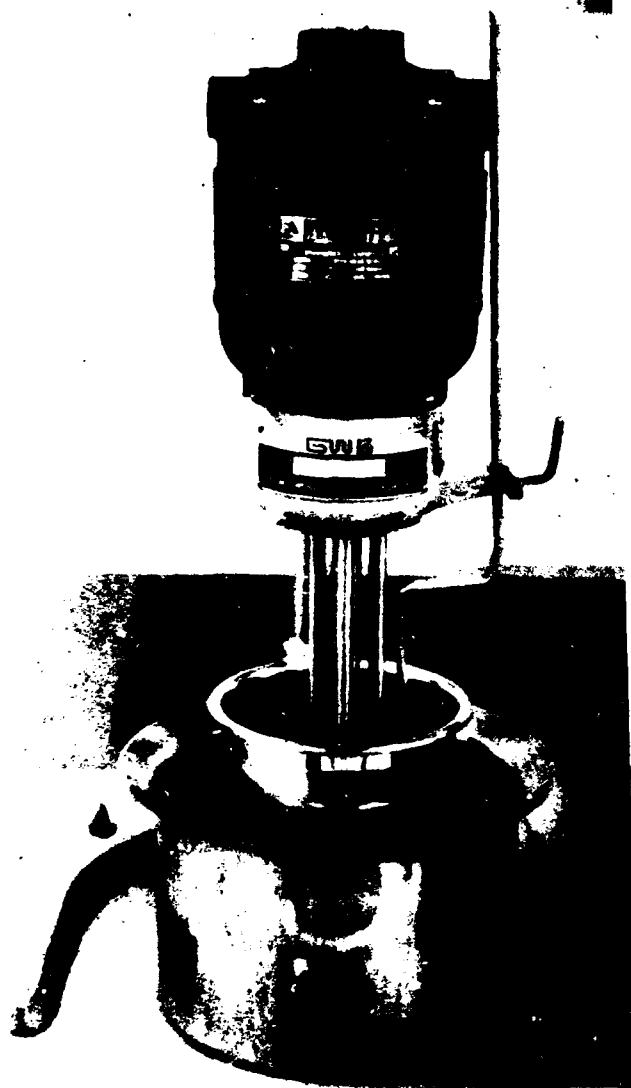
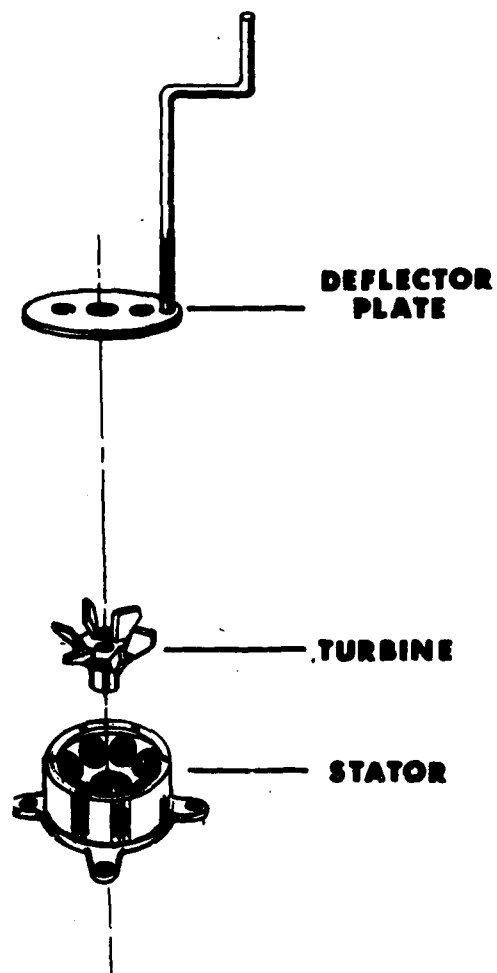


FIGURE 1 -



MIXER HEAD DETAILS

## L-1 HOMOGENIZER MIXER

WITH MIXING VESSEL  
AND ICE BATH

plate. Models are available with batch capacity of 2000 gallons and pipeline (continuous) models capable of 2000 gallons per minute.

Cowles Dissolver - A conventional high shear mixer designed with a drive shaft extending to the base of the mixing vessel. A circular impeller having an arrangement of blades provide intensive mixing. The agitator RPM equals the motor RPM.

High Shear Blender - For short duration mixing and small experimental sample batches of active carbon slurries, a conventional "Waring" type blender was used for convenience. This type mixer was primarily used in testing the water soluble polymer thixotropes.

Viscosity Measurements - The viscosity of the mixtures prepared was measured with a Brookfield Syno-lectric Viscometer fitted with a Helipath Stand. This attachment is especially designed for non-Newtonian viscosity measurements, moving the spindle in a sinusoidal path to avoid tracking and the attendant decrease in shear stress. Results have been very consistent over a range of 100 to 60,000 centipoise depending upon shear rate, i.e., spindle speed.

Settling Tests - A centrifuge was set up to test carbon slurries for settling. Based on previous work with thixotropes, it was determined that centrifuging a loaded thixotropic material at 6 g's for 5 minutes simulates storage of the material for 2 years. A centrifuge having a swing arm radius of 1.1275 feet turning at 125 RPM results in the required 6 g loading. Test Tube samples of the slurry were placed in the holder and tested for the required 5 minutes. From the behavior of the slurry, simulated storage can be determined in a short period of time.

Spray Equipment - An apparatus using nitrogen gas as an expellant was used in experimental sprayings. A measured average pressure of 200 psi was used simulating the standard nitrogen cartridges used in present U.S. Army spraying equipment.

To allow a variety of nozzles to be investigated, an adaptor was machined to allow the sprayer to accept certain types of commercially available nozzles. Nozzles supplied by the Grayco and DeVilbiss Companies were evaluated.

Aluminum Test Panels - Two 24" x 24" aluminum test panels were painted with 1 mil of chromate primer and 1 mil of lusterless, GG, Alkyd Lacquer, as required by the Edgewood specification. Test tabs, one square inch, were used to determine coverage.

## C. Formulation and Experimentation

### 1. Organic Polymer Thixotropes

Data and experience obtained from an earlier thixotropic study was used as a starting point to begin work on this program. This earlier project involved the development of a thixotropic propellant that would suspend particles in the low micron range and would remain completely stable and uniform over a two year period at static storage. This was successfully formulated by a thixotrope with an apparent viscosity of 20,000 centipoise at a shear rate of  $1 \text{ sec}^{-1}$  and 200 centipoise at a shear rate of  $200 \text{ sec}^{-1}$ . This was accomplished using a water soluble resin to form a carboxy vinyl polymer system. Application also required that the material be forced through a small orifice very similar to spraying. Thus a high degree of thixotropy was necessary.

With this data in mind, the initial experiments were made to determine the effects of pretreatment of material, sequence of addition and method, intensity and duration of mixing on the resulting mixture.

Two typical formulations were used in this initial work; one composed of carbon, microcrystalline asbestos, pigment, sodium chromate corrosion inhibitor and carrier (alcohol-water); the second composed of carbon, Carbopol 934 vinyl polymer thixotrope, pigment, sodium chromate and carrier. In some formulations, traces of wetting agent, and triethanolamine as a neutralizer, were used.

A variation in composition, treatment and process was tried. Depending upon the formulation under test, the following general steps apply.

#### Step One - Addition of Thixotropic Agents

When formulating a thixotrope using the polymeric gelling agent, it is necessary to dissolve the powder into the carrier at high shear such as a "Waring" blender will produce. Attempts to add this gelling agent to the combined carbon-pigment slurry results in undissolved particles of Carbopol and inconsistent mixing. On the other hand, Avibest C, microcrystalline asbestos, was found to disperse more evenly and form better mixtures when added to the combined carbon-pigment mixture than when blended into either independently.

#### Step Two - Addition of Carbon and Pigment to the Thixotrope

Best results have been accomplished by thoroughly mixing the carbon and pigment together before adding to the carrier. A number of methods were tried including (1) screening the two components



together through a 40 mesh screen, (2) ball milling for 15 to 30 minutes, and (3) hand mixing with a spatula. Hygroscopic properties of the pigment limit the effect of ball milling as the dry material tends to "pack" causing the ineffective mixing. Either screening or hand mixing thoroughly was proven satisfactory.

#### Step Three - Required Mixing to Form Uniform Slurry

Tests were conducted on standard impeller mixers, high shear Welax types, Cowles dissolvers, and Waring blender type mixers. It was found that short duration (less than 1 hour) high shear mixing, such as that experienced in the Cowles dissolver or Welax mixer forms more stable suspensions than longer mixing at any shear rate. To establish a breakdown point caused by excessive shear mixing, tests have been conducted for several hours resulting in both Carbopol and Avibest C suspensions having been degraded by excessive mixing.

It was found that the consistency of the carbon slurries varied considerably with the type carbon used, i.e., the finer particle sizes formed a very thick paste. With the smaller particle sized carbons, additional carrier was required to keep the mixture sufficiently fluid to complete the formulation. The CWS carbon (1-2 $\mu$  range) wet out easily without additional carrier.

Results obtained in the preliminary evaluation indicated the Carbopol polymer system to be both easier to formulate and more stable after short mixing times than was the Avibest C. Considering ease of manufacturing as a primary objective, it was desired to concentrate efforts in preparing a stable thixotropic slurry using the Carbopol system. Avibest C was later withdrawn from the market and was not available for further evaluation.

#### Step Four - Stabilization of Carbon/Carbopol Thixotropes

Carbopol water-soluble resins are carboxy vinyl polymers of extremely high molecular weight supplied as dry fluffy powders in acid form. This material requires neutralization to develop optimum properties. The solutions gel at a pH of 7-10 and exhibit the following properties.

- Thickening efficiency - very small amounts (less than 1/2%) are often required to give the required viscosity.
- Stability - relatively unaffected by temperature and is not subject to hydrolysis or oxidation.
- Safe and Resistant - used in drugs and cosmetic applications and is not attacked by or will not support mold growth.
- Versatile and Uniform - used in many applications and affords reproducibility.

Carbopol gelling agent is available in three concentration forms, i.e., 934, 940 and 941.

The following tests were performed under identical conditions for comparison. All gels were prepared in a Waring type blender with shear mixing constant at 2 minutes.

Test 1 - 300 ML Solvent (40 methanol/60 water)

1 g Carbopol 934

5 drops triethanolamine

---

Viscosity at  $1 \text{ sec}^{-1}$  and  $77^\circ\text{F}$  - 7,000 cps

Test 2 - 300 ML Solvent

1 g Carbopol 940

5 drops triethanolamine

---

Viscosity at  $1 \text{ sec}^{-1}$  and  $77^\circ\text{F}$  - 16,000 cps

Test 3 - 300 ML Solvent

1 g Carbopol 941

5 drops triethanolamine

---

Viscosity at  $1 \text{ sec}^{-1}$  and  $77^\circ\text{F}$  - 1,000 cps

Because Carbopol 934 exhibits the highest degree of thixotropy (Figure 2) which is advantageous in spray applications, it was selected to develop a stable thixotropic suspension with activated carbon. A typical formulation for this phase of work contained:

CWS Carbon	180 g
Carbopol 934	3 g
1100 Yellow	30 g
Sodium Chromate	3 g
Carrier	950 ML (60/40 water/methanol)
Triethanolamine	3 g

and, in some experiments

HCL 1-3 drops

When activated carbon is chemically basic, mixing the Carbopol-Carbon slurry was hampered by premature gelling of the Carbopol. To prevent this, one of two methods can be employed. First, the carbon slurry was neutralized with hydrochloric acid permitting the blending of the formulation followed by the addition of more triethanolamine which renders the mixture basic again. Results of this technique were somewhat erratic, possibly due to the

absorptivity of the carbon which tends to delay the neutralization of the carbon slurry both with acid and again with triethanolamine. Second, the Carbopol was added to the carrier prior to the addition of the carbon and pigment. After high shear mixing to a uniform slurry, the neutralizer was added to form the gel. This second method proved to be the more effective.

After formulating a variety of suspensions using different concentrations of gelling agents and mixing techniques, the stability study began. Stability of mixtures prepared was checked by static storage for up to eight weeks and by centrifugal tests. As described in the equipment section, centrifugal tests at 6 g's for 5 minutes is roughly equivalent to two years static storage. Accordingly, this test has been applied here. Although static storage time is relatively short, the results are consistent, i.e., those mixtures failing the centrifugal tests have shown settling in 1-6 weeks storage.

The causes and effects of syneresis was briefly investigated. After 18-24 hours storage, unstable and even some apparently stable gels accumulate small quantities of solvent (exudate) on the surface. Attempts were made with this exudate either to prevent its occurring or to possibly affect its recombination.

After numerous experiments, it was discovered that the corrosive inhibitor, sodium chromate, had adverse effects on the carboxy vinyl polymer. By omitting this particular corrosive inhibitor the problem of syneresis was eliminated.

After the formation of Carbopol 934/carbon thixotropes with an apparent viscosity in the 20,000 centipoise range, selected samples were placed in sealed glass containers to test the effects of static storage. After undisturbed storage ranging from 10 to 30 days, viscosity measurements showed a decrease in those samples selected for tests. This unexpected drop in viscosity in most cases resulted in light or "soft" settling of some carbon particles.

Tables I and II lists formulations of Carbopol 934 and 941 selected for static storage tests and the viscosity changes.

Although reconstitution can be easily accomplished by one or two shakes of the container, it is felt that a completely stable slurry could be obtained.

In selecting the Carbopol 934 to begin the investigation, consideration was given to the degree of thixotropy at various shear rates as exhibited in Figure No. 2. This chart gives the viscosity at different shear rates and shows Carbopol 934 to exhibit a greater viscosity drop as the shear increases. This, of course, would

result in a more sprayable thixotrope. As shown by the chart, Carbopol 941 is less affected by increases in shear, thus somewhat less attractive for spray applications, but gives a more stable thixotrope at lower viscosity.

Applying the same techniques as developed for Carbopol 934, formulations were made using Carbopol 941 as the gelling agent. Table II gives results of several formulations using Carbopol 941. As before, various concentrations were stored in sealed glass containers to test the effect of static storage. As this table shows, relatively no changes in viscosity or pH were measured over the thirty day storage period.

Due to the fact that the Carbopol 941 thixotropes are somewhat harder to spray, efforts to develop the lowest possible stable viscosity were pursued. Slurries with measured viscosities of 5,000 centipoise show stability at static storage for 30 days.

#### Results of Organic Polymer Systems as Thickening Agents

After stabilized mixtures of active carbon, pigment, corrosion inhibitor and water-alcohol, with the carboxy vinyl polymer system as the thixotropic agent, had been successfully developed, samples of the carbon/Carbopol suspensions were submitted to Edgewood for gas absorptive tests.

Although this particular system satisfied all the requirements of the Edgewood specification related to settling, permeability, sprayability, etc., results of the agent tests performed at Edgewood showed that the film forming properties of the vinyl polymer gels adversely affect the absorptive properties of the active carbon. As this property of the carbon is essential, organic thixotropes of this type were eliminated from additional study.

A total of seventy-seven experiments were conducted in evaluation of the vinyl polymer thickening system; therefore, tabulation of experiments with the inorganic thixotropic agents will begin with number 78 in this report.

## 2. Inorganic Thixotropes

### a. General Outline of Investigation

Due to the infinite number of possible combinations of materials and mixing equipment, key combinations were selected as shown in Table III. The table represents a general scheme of procedure. Experiment IX will serve to

TABLE I  
STATIC STORAGE TESTS OF  
CARBON/CARBOPOL 934 THIXOTROPES

Formula

CWS Carbon            180 g  
Solvent                950 ml  
Carbopol 934          2.6 g  
TEA                    27 drops    \*47 drops  
1100 Yellow            30 g

Procedure

From Thixotrope,  
Mix carbon/1100,  
Blend into carrier  
Shear

FORMULATION NUMBER	SHEAR TIME WELAX MIXER	pH	STABILITY, VISCOSITY, CPS					
			Imed. After Mix	After 16 hrs	After 10 days	After 20 days	After Reconstitution	
C-1	32 min. @ 25 psi	6.9	21,800	22,700	12,000	12,000 soft settling	10,000	pH 7.2
C-2	32 min. @ 25 psi	6.4	26,000	23,000	17,000	17,000 soft settling	7,000	7.2
C-3	5 min. @ 45 psi 2 min. @ 40 psi 25 min. @ 25 psi	6.7	16,000	16,000	10,000	9,000 settling	3,000	7.2
C-4*	5 min. @ 45 psi 2 min. @ 40 psi 25 min. @ 25 psi	7.0	26,000	24,000	18,000	10,000 soft settling	10,000	7.3
C-5*	5 min. @ 45 psi 2 min. @ 40 psi 15 min. @ 25 psi	7.1	31,000	33,000	23,000	15,000 holding	15,000 holding	7.3
C-6*	5 min. @ 45 psi 2 min. @ 40 psi 25 min. @ 32 psi	7.1	24,000	20,000	17,000	10,000 holding	10,000 holding	7.2

TABLE II

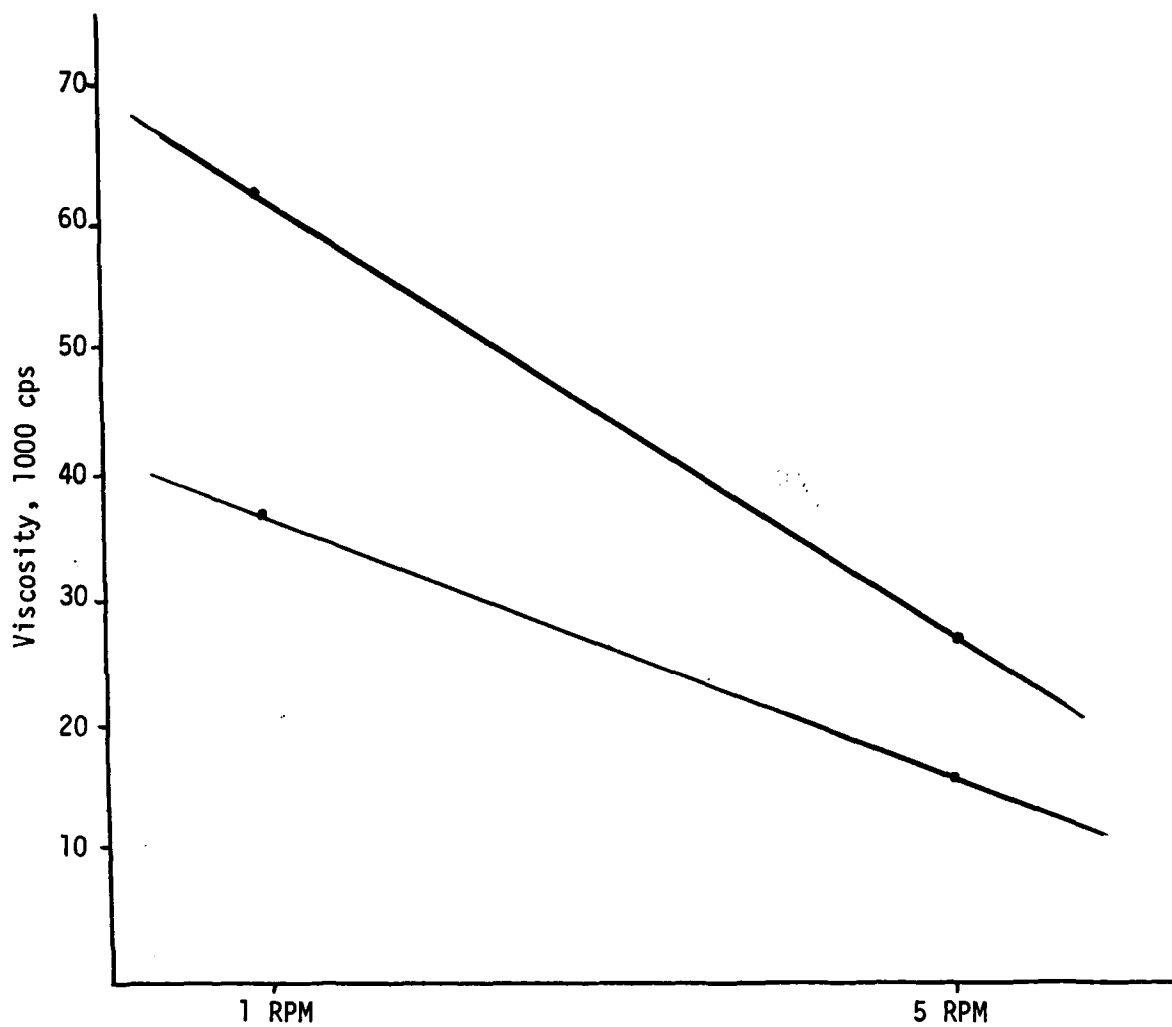
## STATIC STORAGE TEST OF CARBON/CARBOPOL 941 THIXOTROPES

Formula	Procedure
BPL Carbon	Carbon blend into carrier
Solvent	Carbopol blend into carrier
Carbopol 941	Two mixed together
Neutralizer	Shear and neutralize
1100 Yellow	
180 g	
950 ml	
(as shown)	
(as shown)	
30 g	

EXPERIMENT NUMBER	CARBOPOL 941 & NEUTRALIZER	STABILITY, VISCOSITY & pH											
		IMMEDIATELY AFTER MIX		AFTER 24 HRS.		AFTER 48 HRS.		AFTER 3 DAYS		AFTER 5 DAYS		AFTER 30 DAYS	
		cps	pH	cps	pH	cps	pH	cps	pH	cps	pH	cps	pH
C-7	1% & TEA <sup>1</sup>	14,000	7.1	14,000	7.2	14,000	7.1	14,000	7.1	14,000	7.1	14,000	7.1
C-8	1% & TEA <sup>2</sup>	7,000	7.2	7,000	7.0	7,000	7.0	8,000	7.1	8,000	7.1	8,000	7.1
C-9	1% & DIISO <sup>3</sup>	12,000	7.0	12,000	6.9	12,000	6.9	12,500	6.0	12,500	6.9	12,500	6.9
C-10	.50% & TEA	7,500	7.2	7,500	7.3	7,500	7.2	7,500	7.2	7,000	7.2	7,000	7.2
C-11	.33% & TEA	6,000	7.3	6,000	7.3	5,500	7.2	5,500	7.2	5,500	7.2	5,500	7.2
C-12	.15% & TEA	800	7.5	600	7.5								
		← Settling →											

## NOTES:

<sup>1</sup> 1% Carbopol 941 based on solvent with equal amount of Triethanolamine.<sup>2</sup> Same quantities of Carbopol 941 and TEA with double amount of shear as No. C-7.<sup>3</sup> Diisopropanolamine



Shear Rate  
Brookfield Helipath Spindle Speed

FIGURE 2  
VISCOSITY VS. SHEAR RATE IN  
MEASURING THE DEGREE OF THIXOTROPY

evaluate types of mixing equipment for a preliminary selection based on uniformity, stability and ease of mixing of the slurries.

Experiments 2X, 3X, and 4X will serve to evaluate the basic stability of the carbon blacks. Formulas 5X, 6X, and 7X will show the effect of thixotroping agents on a particular carbon black. Experiments 8X, 9X, and 10X will aid in evaluating dispersants.

Generally, materials will be eliminated from the program based upon their performance when put into suspension, i.e. ease of mixing, stability, effect on viscosity, sprayability and more important, gas absorptive properties.

Viscosities are being taken with the Brookfield Viscometer, Model RVT, utilizing the Helipath attachment. Readings are also being taken with a standard #4 spindle at 10 and 20 rpm. The viscometer is allowed to stabilize for 10 revolutions before readings are taken.

Formulations are being stored as Parts A and B. Part A is being observed for stability at undisturbed storage. Part B is being reconstituted prior to checking the viscosity after 1, 7, 14, 30 and 90 days.

b. Selection of Optimum Mixing Equipment with a Standard Formulation

To measure the mixing efficiency of the different pieces of equipment, a standard formulation was selected. The formulation was as follows:

<u>Ingredient</u>	<u>% Formula Wt.</u>	<u>Actual Wt.</u>
CWS Carbon	25.0	300 g
Bentonite	.5	6 g
DAXAD 11	1.0	12 g
1100 Yellow Pigment	5.0	60 g
Sodium Chromate	.5	6 g
Methanol-Water	68.0	756 ml

Slurries were prepared by mixing for 30, 60, 90, 120 and 180 minute intervals using the homogenizer and Cowles Dissolver. Friction-heating resulted in the need to utilize an ice bath to maintain a constant temperature below 70°F. Tables IV and V give results of these preparations. Slurries were also prepared by ball milling for 48 hours. These results are shown in Table VI.



TABLE III  
PREPARATION OF CARBON SUSPENSIONS

TYPICAL EXPERIMENT NUMBER		1X	2X	3X	4X	5X	6X	7X	8X	9X	10X
Based on 100% by Weight (% Solvent = 100% - % Formula Weight)											
Carbon	BPL (CWS)	25				25	25	25	25	25	25
Thixotrope	UU		25								
	ACC			25							
	KC				25						
	Bentonite	.5	.5	.5	.5	.5	.5	.5			
Dispersants	RG-244								.5		
	COK-84									1	
	Aviarnide-6-P Nylon										1
	DAXAD 11	1	1	1	1				1	1	1
Pigments	Marasperse CB					1					
	Darvan No. 2						1				
	Nopcosant K							1			
	1100 Yellow	5	5	5	5	5	5	5	5	5	5
Corrosive Inhibitor	Phthalocyanine Green										
	Cadmium Yellow										
	Sodium Chromate	.5	.5	.5	.5	.5	.5	.5	.5	.5	.5
	Sodium Silicate										

As shown by Tables IV and V, after 60 minute mixing time the homogenizer consistently gave slurries with higher viscosities than does the Cowles dissolver and more closely approaches the uniformity of those slurries prepared by ball milling (Table VI).

As each of the tables show, the viscosity of most formulations increases during storage. The reason for this increase is discussed later in this report. At this point, two possible reasons are that (1) during mixing only a small portion of the absorptive power of the carbon may be taken up by the solvent. During storage each particle may continue to absorb the solvent thus increasing the viscosity. Second, the fact that the slurries subjected to the least amount of mixing had the greatest increase in viscosity, it could be that insufficient mixing has produced a poorly structured bentonite lattice. During storage this weak lattice tends to collapse causing viscosity increases.

After evaluating the three different mixing techniques shown in Tables IV, V, and VI, the homogenizer type mixer was found to be superior from the standpoint of uniformity and ease of mixing. Therefore, it was selected as the mixing technique to be used throughout the remaining portions of the program.

c. Evaluation of the Different Based Carbons with the Key Ingredients

(1) CWS Carbon - Coal Based

The coal based activated carbon used in this evaluation was manufactured by the Pittsburgh Activated Carbon Company. The carbon supplied to Brunswick by Edgewood Arsenal had an average particle size of 1.5  $\mu$  and a pH value of 10.0 in aqueous solution. The surface area measured 1000-1100 sq. meters/gram with a  $\text{CCL}_4$  activity of 65%.

The different thixotropic and dispensing agents, shown in Section II-A, were first evaluated for effects on aqueous slurries of CWS activated carbon. Formulations were prepared with each thixotrope, i.e., bentonite, asbestos, pyrogenic silica and microcrystalline nylon, and are shown in Table VII.

Experiments 79, 83, 87, and 87-A show effects of each of the thixotropes when using Darvan No. 1 as the dispersing agent. As this series of experiments show, bentonite was the most efficient thixotropic agent in that it gave the highest, most stable viscosity. Experiments 115, 116, 117 and 118 were performed under the same condition substituting DAXAD 11 as the dispersant. Results again show bentonite to be the most efficient thixotropic agent by giving the highest viscosity with the other additives being held constant.

TABLE IV  
CARBON SUSPENSION PREPARED WITH THE L-1 HOMOGENIZER

Formulation No.	1-78	1-79	1-80	1-81	1-92
Mixing Time, Minutes	After 30	After 60	After 90	After 120	After 180
IMED. Helipath (1)	12,000	9,000	8,000	11,000	11,000
AFTER #4* @ 10 RPM (2)	9,000	6,800	6,000	8,400	8,200
MIX #4 @ 20 RPM	5,100	3,400	3,100	4,100	4,150
AFTER Helipath	15,000	12,000	10,000	10,000	11,000
24 #4 @ 10 RPM	14,000	9,000	7,600	7,500	8,700
HOURS #4 @ 20 RPM	7,500	4,700	3,900	3,800	4,200
AFTER Helipath	16,500	16,000	13,000	15,000	14,000
7 #4 @ 10 RPM	15,000	12,800	10,800	10,000	12,000
DAYS #4 @ 20 RPM	6,650	6,200	5,450	5,450	5,750
Solvent Content, %	58.7	63.0	59.0	61.3	58.2
pH	10.5	10.5	10.4	10.4	10.4

(1) Brookfield Viscometer Model RVT  
with special Helipath attachment  
for measuring thixotropic materials

(2) Brookfield Viscometer Model RVT  
with Standard #4 Spindle

TABLE V  
CARBON SUSPENSIONS PREPARED WITH A COMLES DISSOLVER

Formulation No.	1-84	1-84	1-85	1-93	1-94
Mixing Time, Minutes	After 30	After 60	After 90	After 120	After 180
IMEL. Helipath	Unmixed	5,000	4,000	7,800	7,000
AFTER #4 @ 10 RPM		4,800	3,900	6,000	5,500
MIX #4 @ 20 RPM		2,500	2,000	3,000	2,750
AFTER Helipath	Unmixed	7,100	6,250	7,900	7,000
24 #4 @ 10 RPM		5,000	4,900	7,000	6,000
HOURS #4 @ 20 RPM		2,700	2,900	3,500	3,050
AFTER Helipath		9,000	8,000	12,000	10,000
7 #4 @ 10 RPM		2,700	7,600	9,700	8,000
DAYS #4 @ 20 RPM		3,100	3,900	4,850	4,000
Solvent Content, %		58.7	59.8	61.2	60.4
pH		10.3	10.3	10.4	10.3

TABLE VI  
CARBON SUSPENSIONS PREPARED WITH A BALL MILL

Formulation No.	1-91	1-82	1-86
Mixing Time, Hours	48	48	48
IMED. Helipath	25,000	14,000	17,000
AFTER #4 @ 10 RPM	19,800	12,300	15,600
MIX #4 @ 20 RPM	10,300	7,200	8,400
AFTER Helipath	25,000	14,000	17,000
24 #4 @ 10 RPM	19,200	12,400	13,800
HOURS #4 @ 20 RPM	10,000	7,000	7,500
AFTER Helipath	22,000	15,600	18,000
7 #4 @ 10 RPM	18,600	14,000	16,300
DAYS #4 @ 20 RPM	4,800	8,000	8,450
Solvent Content, %	57.7	57.3	57.2
pH	10.4	10.7	9.7

It was concluded from these two series of experiments that bentonite is a superior thixotropic agent to either asbestos, pyrogenic silica or microcrystalline nylon when using the CWS coal based carbon.

After selecting bentonite as the thixotrope agent for CWS carbon, different dispersing agents, DAXAD 11, Darvan No. 2, Marasperse CB, and Nopcosant K were evaluated for their effectiveness in lowering the viscosity. Experiments 115, 119, 120, and 121 (Table VIII) show initial evaluations of the dispersants. By assuming the lower viscosity indicates a better dispersion of carbon particles, experiment 120, using Marasperse CB dispersant, was selected for additional evaluation.

By analyzing experiments 134, 135, 137, and 141 (Table IX) it can be seen that carbon loadings up to 40% (Exp. 137) are possible using Marasperse CB, however, these slurries become glue-like in texture with a significant drop in viscosity during storage. Experiments 144, 145, and 147 were conducted to maximize carbon loading with a more stable viscosity. Experiment 145 using DAXAD 11 gave both a carbon content of 31.7% and more stable viscosity during storage. Therefore, DAXAD 11 was chosen as the superior dispersant.

After selecting bentonite as the most efficient thixotrope and DAXAD 11 as the dispersing agent, the next step was to determine the optimum percent concentrations of DAXAD 11. This was done by conducting the experiments shown in Table X. The results are also shown charted in Figure 3. Experiments 115, 149, 150, and 151 were conducted with a 300 gram CWS carbon loading (~25%) with 12, 24, 36, and 48 grams (~ 1, 2, 3, & 4%) of DAXAD 11 dispersant.

Experiments 152, 145, 153, and 154 contained 395 grams (31%) carbon loading. As the results show, there is a significant difference in reaction of the DAXAD 11 to the percent carbon loadings. This data indicates optimum to be 2% DAXAD 11 dispersant with each carbon loading.

The next series of experiments were prepared to test the effect of corrosion inhibitors. Experiments 145, 172, and 176, shown in Table XI, give the resulting viscosities of suspension prepared with sodium chromate and sodium silicate inhibitors. Both Experiments 176 and 172, containing .48% and .96% sodium silicate inhibitor, show a drop in viscosity with eventual settling. Sodium silicate did show a slight increase in pH over the sodium chromate. Based on stability of the suspensions, sodium chromate is selected as the better corrosion inhibitor.

Experiments 145, 184, 185, 188, and 201, shown in Table XII, were conducted to evaluate the effect of various mixing times using the L-1 Laboratory Homogenizer. As the results show, each increased mixing time produced a higher viscosity, however no apparent stabilization of the viscosities was achieved. It was concluded that additional mixing time beyond thirty (30) minutes does not improve the stability of the CWS suspensions. The solvent content of Experiments 145 and 201 measured 55.8% and 55.5% respectively, therefore solvent evaporation during mixing did not produce the increased viscosities.

Various types of pigments were evaluated to produce an olive green color. Cadmium lithopone yellows, oranges and phthalocyanine green pigments supplied by Harshaw Chemical Company were added to CWS suspension on concentrations up to 6.3%. As Table XIII shows, Experiments 191 with the Azo yellow and 193 with the cadmium lithopone gave considerable increases in viscosity without the desired olive green color. A 6.3% concentration of phthalocyanine green remained black. The Mapico 1100 yellow, iron oxide, pigment did give the desired olive green at a 4.8% level, therefore was selected over the other pigments tested.

After making final selections of a thixotrope, dispersant, pigment and corrosion inhibitor for use with the CWS carbon, some adjustments were made in solvent content. With a carbon content of 31.60% and a solvent content of 60.71% the resulting viscosity was in the 25,000 centipoise range. As can be seen by Spray Test No. 12, the coverage over the test panel was in the 20 mg/cm<sup>2</sup> range indicating the viscosity was too high for effective spraying. Also the resulting spray gave a spattered, non-uniform coating again indicating the material to be thick. The solvent content was then increased to 63.03% (reducing the carbon to 29.59%) which resulted in a viscosity in the 12-14,000 centipoise range. Slurries in this viscosity range can be effectively sprayed.

A typical formulation for CWS carbon used both for testing sprayability and adhesion would be as follows:

<u>Ingredient</u>	<u>Actual Weight</u>	<u>Percent Formula Wt.</u>
Carbon: CWS Carbon	385 g	29.59
Thixotrope: Bentonite	6 g	.46
Dispersant: DAXAD 11	24 g	1.84
Pigment: 1100 Yellow	60 g	4.61
Corrosion Inhibitor: Sodium Chromate	6 g	.46
Solvent: (60/40 Water-Methanol)	820 ml	63.03
Mixing Time: Thirty (30) minutes using the L-1 Laboratory Homogenizer		
Typical Viscosity at 24 hours	14,000 cps	Helipath
after mixing	11,000 cps	#4 @ 10 RPM
	5,500 cps	#4 @ 20 RPM
pH Value	10.4	
Carbon Particle Size	1-2 $\mu$	(avg.)



TABLE VII

## CWS CARBON - THIXOTROPE EVALUATION

FORMULATION NO.	79	83	87	87-A	115	116	117	118
COMPOSITION BY/WT.	Actual Wt.							
Carbon CWS - Coal Based Carbon	300 g	300 g	300 g	300 g	300 g	300 g	300 g	300 g
Thixotrope (1) Bentonite	6 g				6 g			
(2) RG-244		6 g				6 g		
(3) COK-84			6 g				6 g	
(4) Nylon-6				6 g				6 g
Dispersant (1) DAXAD 11					12 g	12 g	12 g	12 g
(2) Darvan #1	12 g	12 g	12 g	12 g				
(3) Marasperse CB								
(4) Nopcosant K								
Pigment 1100 Yellow	60 g	60 g	60 g	60 g	60 g	60 g	60 g	60 g
Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g	6 g	6 g	6 g	6 g	6 g	6 g	6 g
(2) $\text{Na}_2\text{S}_2\text{O}_3$								
Solvent, Water/Alcohol 60/40 By Vol.	756 ML	756 ML	756 ML	756 ML	756 ML	756 ML	756 ML	756 ML
MIXING TECHNIQUE: Homogenizer, min.	60	60	60	60	30	30	30	30

## CHARACTERISTICS

## Viscosity, CP

Imed.	Helipath "C"	9,000	2,000	3,000	1,000	7,000	1,800	3,000	1,500
After	#4 @ 10 RPM	6,800	1,900	2,800	500	5,700	1,100	2,440	1,000
Mix	#4 @ 20 RPM	3,400	1,050	1,500	300	2,875	575	1,325	500
After	Helipath "C"	12,000	2,800	4,000	500	10,000	2,000	4,000	2,500
24	#4 @ 10 RPM	9,000	2,200	3,600	200	7,900	1,200	3,350	1,900
Hours	#4 @ 20 RPM	4,700	1,100	1,900	150	4,000	600	1,800	950
After	Helipath "C"	16,000	3,600	6,700		14,000	2,500	6,500	3,000
7	#4 @ 10 RPM	12,000	2,900	5,600		13,700	1,600	5,000	2,100
Days	#4 @ 20 RPM	6,200	1,300	2,750		4,850	775	2,650	1,000
After	Helipath "C"	17,000	2,000	7,000		14,000	2,900	6,800	2,500
14	#4 @ 10 RPM	12,500	1,400	5,700		10,200	1,500	5,350	2,300
Days	#4 @ 20 RPM	8,150	750	2,800		5,200	800	2,900	1,175
After	Helipath "C"	16,800	3,700	8,000		14,000	2,000	7,000	2,900
30	#4 @ 10 RPM	13,000	2,100	5,800		11,300	1,100	5,000	2,150
Days	#4 @ 20 RPM	6,800	1,125	2,800		5,550	600	2,550	1,000
pH		10.5	10.3	10.2		9.7	9.5	9.9	9.8

NOTES:

TABLE VII

## CWS CARBON - THIXOTROPE EVALUATION

FORMULATION NO.	79	83	87	87-A	115	116	117	118
COMPOSITION BY/WT.	Actual Wt.							
Carbon CWS - Coal Based Carbon	300 g	300 g	300 g	300 g	300 g	300 g	300 g	300 g
Thixotrope (1) Bentonite	6 g				6 g			
(2) RG-244		6 g				6 g		
(3) COK-84			6 g				6 g	
(4) Nylon-6				6 g				6 g
Dispersant (1) DAXAD 11					12 g	12 g	12 g	12 g
(2) Darvan #1	12 g	12 g	12 g	12 g				
(3) Marasperse CB								
(4) Nopcosant K								
Pigment 1100 Yellow	60 g	60 g	60 g	60 g	60 g	60 g	60 g	60 g
Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g	6 g	6 g	6 g	6 g	6 g	6 g	6 g
(2) $\text{Na}_2\text{S}_2\text{O}_3$								
Solvent, Water/Alcohol 60/40 By Vol.	756 ML	756 ML	756 ML	756 ML	756 ML	756 ML	756 ML	756 ML
MIXING TECHNIQUE: Homogenizer, min.	60	60	60	60	30	30	30	30

## CHARACTERISTICS

## Viscosity, CP

Imed.	Helipath "C"	9,000	2,000	3,000	1,000	7,000	1,800	3,000	1,500
After	#4 @ 10 RPM	6,800	1,900	2,800	500	5,700	1,100	2,440	1,000
Mix	#4 @ 20 RPM	3,400	1,050	1,500	300	2,875	575	1,325	500
After	Helipath "C"	12,000	2,800	4,000	500	10,000	2,000	4,000	2,500
24	#4 @ 10 RPM	9,000	2,200	3,600	200	7,900	1,200	3,350	1,900
Hours	#4 @ 20 RPM	4,700	1,100	1,900	150	4,000	600	1,800	950
After	Helipath "C"	16,000	3,600	6,700		14,000	2,500	6,500	3,000
7	#4 @ 10 RPM	12,000	2,900	5,600		13,700	1,600	5,000	2,100
Days	#4 @ 20 RPM	6,200	1,300	2,750		4,850	775	2,650	1,000
After	Helipath "C"	17,000	2,000	7,000		14,000	2,900	6,800	2,500
14	#4 @ 10 RPM	12,500	1,400	5,700		10,200	1,500	5,350	2,300
Days	#4 @ 20 RPM	8,150	750	2,800		5,200	800	2,900	1,175
After	Helipath "C"	16,800	3,700	8,000		14,000	2,000	7,000	2,900
30	#4 @ 10 RPM	13,000	2,100	5,800		11,300	1,100	5,000	2,150
Days	#4 @ 20 RPM	6,800	1,125	2,800		5,550	600	2,550	1,000
pH		10.5	10.3	10.2		9.7	9.5	9.9	9.8

NOTES:

TABLE VIII

## CWS CARBON - DISPERSANT EVALUATION

FORMULATION NO.	115	119	120	121				
COMPOSITION BY/WT.			Actual Wt.					
Carbon CWS - Coal Based Carbon	300 g	300 g	300* g	300 g				
Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g				
(2)								
(3)								
(4)								
Dispersant (1) DAXAD 11	12 g							
(2) Darvan No. 2		12 g						
(3) Marasperse CB			12 g					
(4) Nopcosant K				12 g				
Pigment 1100 Yellow	60 g	60 g	60 g	60 g				
Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g	6 g	6 g	6 g				
(2)								
Solvent, Water/Alcohol 60/40 By Vol.	756 ML	756 ML	756 ML	756 ML				
MIXING TECHNIQUE: Homogenizer, min.	30	30	30	30				

## CHARACTERISTICS

## Viscosity, CP

Imed.	Helipath "C"	7,000	3,500	1,100	11,000			
After	#4 @ 10 RPM	5,700	2,200	650	9,600			
Mix	#4 @ 20 RPM	2,875	1,225	400	5,675			
After	Helipath "C"	10,000	1,500	1,000	9,400			
24	#4 @ 10 RPM	7,900	900	600	8,500			
Hours	#4 @ 20 RPM	4,000	500	350	4,550			
After	Helipath "C"	14,000	1,000	1,800	7,500			
7	#4 @ 10 RPM	13,700	450	1,000	7,250			
Days	#4 @ 20 RPM	4,850	300	550	3,875			
After	Helipath "C"	14,000	1,000	1,800	7,300			
14	#4 @ 10 RPM	10,200	400	1,000	6,800			
Days	#4 @ 20 RPM	5,200	250	550	3,675			
After	Helipath "C"	14,000		1,500	6,800			
30	#4 @ 10 RPM	11,300	discarded too thin	750	6,000			
Days	#4 @ 20 RPM	5,550		425	3,325			
pH		9.7	9.5	9.6	9.8			

NOTES: \*MOST EFFICIENT DISPERSANT

TABLE IX

## CWS CARBON - DISPERSANT EVALUATION

FORMULATION NO.	134	135	137	141	144	145	147
COMPOSITION BY/WT.	Actual Wt.						
Carbon CWS - Coal Based Carbon	425 g	450 g	500*g	450 g	430 g	395 g	325 g
Thixotrope (1) Bentonite	6 g	6 g	6 g	9 g	6 g	6 g	6 g
(2)							
(3)							
(4)							
Dispersant (1) Marasperse CB	12+12 g	24 g	24 g	24 g			
(2) Darvan No. 2					24 g		
(3) DAXAD 11						24 g	
(4) Nopcosant K							24 g
Pigment 1100 Yellow	60 g	60 g	60 g	60 g	60 g	60 g	60 g
Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g	6 g	6 g	6 g	6 g	6 g	6 g
(2)							
Solvent, Water/Alcohol 60/40 By Vol.	756 ML	756 ML	756 ML	756 ML	756 ML	756 ML	756 ML
MIXING TECHNIQUE: Homogenizer, min.	30+30	30	30	30	30	30	30

## CHARACTERISTICS

Viscosity, CP		(34,000)						
Imed.	Helipath "C"	10,000	6,000	15,000	15,000	14,500	16,500	11,000
After	#4 @ 10 RPM	5,400	2,800	6,300	6,000	10,250	13,650	10,300
Mix	#4 @ 20 RPM	3,350	1,900	4,750	3,950	6,350	6,650	5,450
After	Helipath "C"	5,000	2,000	12,000	7,000	6,000	16,000	10,000
24	#4 @ 10 RPM	1,850	1,100	4,900	2,900	3,725	13,000	9,200
Hours	#4 @ 20 RPM	1,200	775	3,650	1,950	2,500	6,325	4,900
After	Helipath "C"	3,000	1,500	8,000	5,000	3,200	23,500	9,000
7	#4 @ 10 RPM	1,300	900	2,900	2,100	1,900	17,600	8,200
Days	#4 @ 20 RPM	900	700	2,225	1,400	1,300	8,650	4,500
After	Helipath "C"	2,000	1,200	5,000	4,000	2,800	25,000	8,800
14	#4 @ 10 RPM	1,000	800	2,200	1,625	1,400	19,600	7,700
Days	#4 @ 20 RPM	675	600	1,700	1,125	950	9,800	4,200
After	Helipath "C"	1,000	900	3,000	2,000	2,000	28,000	8,000
30	#4 @ 10 RPM	700	600	2,000	1,150	1,200	22,000	6,300
Days	#4 @ 20 RPM	500	450	1,650	850	850	11,100	3,500
pH		10.1	10.6	10.1	10.2	9.6	10.0	10.0

NOTES:

\*MIXTURE BECAME GLUE-LIKE WITH 500 g CARBON

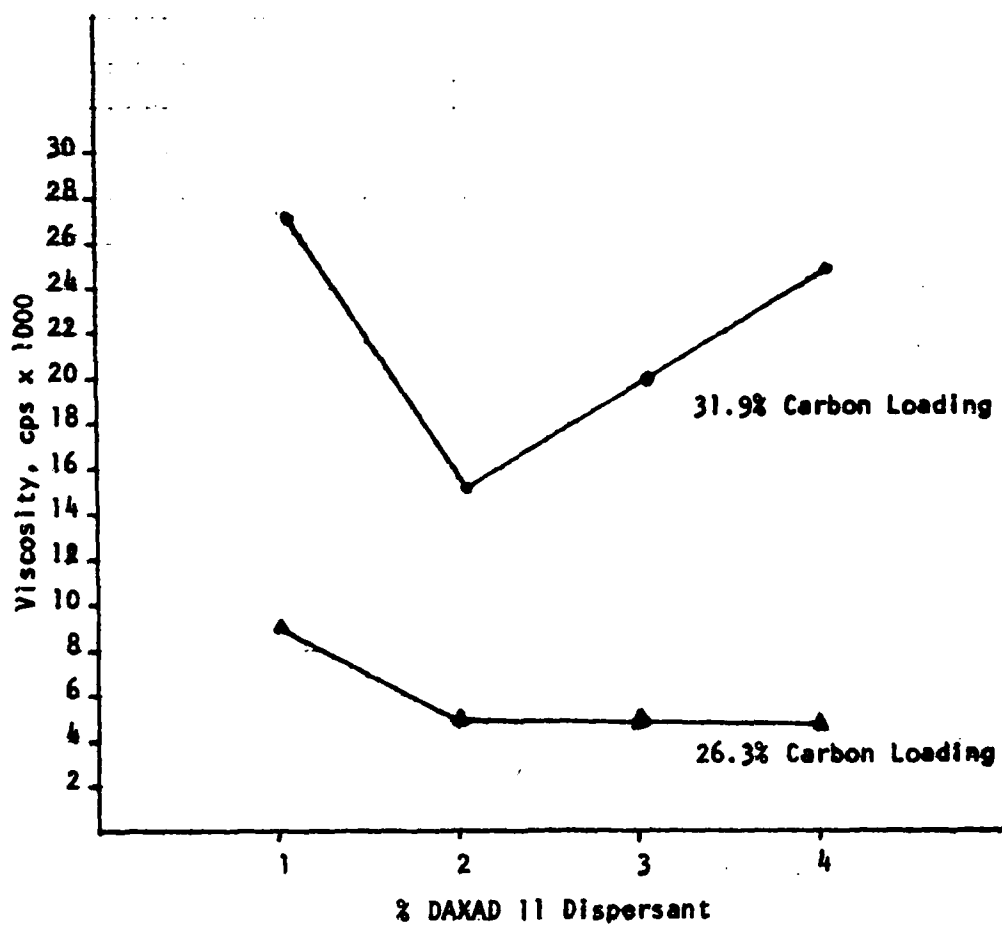


FIGURE 3 - EFFECT OF % DISPERSANT ON CWS CARBON SLURRIES

TABLE X

## CWS CARBON - PERCENT DISPERSANT EVALUATION

FORMULATION NO.	115	149	150	151	152	145	153	154
COMPOSITION BY/WT.	Actual Wt.							
Carbon CWS	300 g	300 g	300 g	300 g	395 g	395 g	395 g	395 g
Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g	6 g	6 g	6 g	6 g
(2)								
(3)								
(4)								
Dispersant (1) DAXAD 11	12 g	24 g	36 g	48 g	12 g	24 g	36 g	48 g
(2)								
(3)								
(4)								
Pigment 1100 Yellow	60 g	60 g	60 g	60 g	60 g	60 g	60 g	60 g
Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g	6 g	6 g	6 g	6 g	6 g	6 g	6 g
(2)								
Solvent, Water/Alcohol 60/40 By Vol.	756 ML	756 ML	756 ML	756 ML	756 ML	756 ML	756 ML	756 ML
MIXING TECHNIQUE: Homogenizer, min.	30	30	30	30	30	30	30	30

## CHARACTERISTICS

## Viscosity, CP

Imed.	Helipath "C"	7,000	4,000	5,000	5,500	25,000	16,500	18,000	26,000
After	#4 @ 10 RPM	5,700	3,000	3,600	3,850	22,900	13,650	13,200	19,800
Mix	#4 @ 20 RPM	2,875	1,575	1,750	1,875	11,450	6,650	6,850	9,700
After	Helipath "C"	10,000	6,000	6,000	5,900	28,000	16,000	21,500	24,000
24	#4 @ 10 RPM	7,900	4,150	4,200	4,300	26,400	13,000	16,400	17,400
Hours	#4 @ 20 RPM	4,000	2,050	2,100	2,140	13,800	6,325	8,100	8,500
After	Helipath "C"								
7	#4 @ 10 RPM								
Days	#4 @ 20 RPM								
After	Helipath "C"								
14	#4 @ 10 RPM								
Days	#4 @ 20 RPM								
After	Helipath "C"								
30	#4 @ 10 RPM								
Days	#4 @ 20 RPM								
pH									

TABLE XI

## CWS CARBON - CORROSION INHIBITOR

FORMULATION NO.	145	176	172					
COMPOSITION BY/WT.				Actual Wt.				
Carbon CWS - Coal Based Carbon	395 g	395 g	395 g					
Thixotrope (1) Bentonite	6 g	6 g	6 g					
(2)								
(3)								
(4)								
Dispersant (1) DAXAD 11	24 g	24 g	24 g					
(2)								
(3)								
(4)								
Pigment 1100 Yellow	60 g	60 g	60 g					
Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g							
(2) $\text{Na}_2\text{S}_2\text{O}_3$		6 g	12 g					
Solvent, Water/Alcohol 60/40 By Vol.	756 ml	756 ml	756 ml					
MIXING TECHNIQUE Homogenizer, minutes	30	30	30					

## CHARACTERISTICS

## Viscosity, CP

Imed.	Helipath "C"	16,500	4,000	11,000				
After	#4 @ 10 RPM	13,650	2,600	6,500				
Mix	#4 @ 20 RPM	6,650	1,600	3,425				
After	Helipath "C"	16,000	1,000	1,000				
24	#4 @ 10 RPM	13,000	1,100	1,900				
Hours	#4 @ 20 RPM	6,325	700	1,100				
After	Helipath "C"	23,500	< 500	< 500				
7	#4 @ 10 RPM	17,600	~ 300	~ 300				
Days	#4 @ 20 RPM	8,650	~ 250	~ 250				
After	Helipath "C"	25,000						
14	#4 @ 10 RPM	19,600						
Days	#4 @ 20 RPM	9,800	Thin Settling	Very Thin Settling				
After	Helipath "C"	28,000						
30	#4 @ 10 RPM	22,000						
Days	#4 @ 20 RPM	11,100						
pH		10.0	10.7	10.8				

TABLE XII

## CWS CARBON - MIXING TIME STUDY

FORMULATION NO.	145	184	185	188	201
COMPOSITION BY/WT.	Actual Wt.				
Carbon CWS - Coal Based Carbon	395 g	395 g	395 g	395 g	395 g
Thixotrope (1) Bentonite					
(2)					
(3)					
(4)					
Dispersant (1) DAXAD 11	24 g	24 g	24 g	24 g	24 g
(2)					
(3)					
(4)					
Pigment 1100 Yellow	60 g	60 g	60 g	60 g	60 g
Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g	6 g	6 g	6 g	6 g
(2)					
Solvent, Water/Alcohol 60/40 By Vol., ml	756	756	756	756	756
MIXING TECHNIQUE: Homogenizer, minutes	30	60	90	120	180

## CHARACTERISTICS

## Viscosity, CP

Imed.	Helipath "C"	16,500	19,000	20,000	28,000	45,000
After	#4 @ 10 RPM	13,650	14,300	15,300	20,200	28,000
Mix	#4 @ 20 RPM	6,650	7,100	7,625	9,900	14,400
After	Helipath "C"	16,000	29,000	28,000	27,000	48,000
24	#4 @ 10 RPM	13,000	19,800	18,000	19,000	32,200
Hours	#4 @ 20 RPM	6,325	9,700	9,100	9,650	16,400
After	Helipath "C"	23,500	34,000	32,000	41,000	53,000
7	#4 @ 10 RPM	17,600	25,100	23,200	29,400	35,600
Days	#4 @ 20 RPM	8,650	12,500	12,000	15,100	18,000
After	Helipath "C"	25,000	36,000	34,000	42,000	60,000
14	#4 @ 10 RPM	19,600	26,200	25,000	33,200	32,500
Days	#4 @ 20 RPM	9,800	12,800	12,200	16,800	19,000
After	Helipath "C"	28,000	34,000	34,000	45,000	57,000
30	#4 @ 10 RPM	22,000	26,400	25,600	33,200	27,500
Days	#4 @ 20 RPM	11,100	13,700	13,500	17,500	18,000
pH		10.0	10.0	10.1	10.0	10.0
NOTES:	Solvent Content, %	55.8				55.5



TABLE XIII

## CWS CARBON - PIGMENT EVALUATION

FORMULATION NO.	145	191	192	193				
COMPOSITION BY/WT.								
			Actual Wt.					
Carbon CWS - Coal Based Carbon	395 g	395 g	395 g	395 g				
Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g				
(2)								
(3)								
(4)								
Pigment (1) 1100 Yellow	60 g							
(2) Azo Yellow		80 g						
(3) Phthalocyanine Green*			80 g					
(4) Cad Yellow/Cad Orange				56/5 g				
Dispersant DAXAD 11	24 g	24 g	24 g	24 g				
Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g	6 g	6 g	6 g				
(2)								
Solvent, Water/Alcohol 60/40 By Vol., ml	756	756	756	756				
MIXING TECHNIQUE	30	30	30	30				

## CHARACTERISTICS

Viscosity, Cp

Imed. After Mix	Helipath "C"	16,500	42,000	3,500	46,000				
	#4 @ 10 RPM	13,600	20,400	1,200	29,800				
	#4 @ 20 RPM	6,650	10,300	750	16,200				
After 24 Hours	Helipath "C"	16,000	/	/	/				
	#4 @ 10 RPM	13,000	/	/	/				
	#4 @ 20 RPM	6,325	/	/	/				
	Color,	Olive	Dark		Medium				
	Wet	Green	Green	Black	Green				
	Color,	Olive	Green		Dark				
	Dry	Green	Black	Black	Green				
pH									

**NOTE:**

\*Supplied by Harshaw Chemical in aqueous suspension

(2) Darco KB Carbon - Wood Based

The wood based carbon is manufactured by the Atlas Chemical Company and is supplied with an average particle size of  $1.2 \mu$ . The pH value in aqueous solution is approximately 5.2 with a surface area of 600-700 square meters/gram and a  $\text{CCl}_4$  activity of 84%.

In evaluating Darco KB carbon, the same general plan of experimentation was used as with the CWS carbon. Experiments 95, 96, 97, 103, 104, and 106, shown on Table XIV, gives results when the carbon is combined with each of the thixotropic agents. After solvent adjustment was made (Exp. 95, 96, and 97) to keep the slurry sufficiently fluid to mix, slurries were prepared with each of the thixotropes using DAXAD 11 as the dispersant. By analyzing Experiments 97, 103, 104, and 106, it can be concluded that bentonite was the most efficient thixotropic agent in that it gave the highest workable viscosity. Although RG-244 asbestos, shown in Experiment 103, gave a very high viscosity, in order to prepare a mixable slurry it would be necessary to (1) lower the carbon content below 24%, (2) lower the thixotrope below 1/2%, which would not be a sufficient amount to hold the carbon particles in suspension, or (3) raise the solvent content, all of which are undesirable.

It was concluded from this series of experiments that bentonite is the superior thixotropic agent with Darco KB wood based carbon.

In evaluating dispersants with the Darco KB-bentonite system, slurries were prepared as shown in Table XV. It can be seen that Darvan No. 2 give the lowest viscosity, indicating the best dispersion of carbon particles. Experiment 148 was conducted using a cationic dispersant, Ammonyx 856, but the data shows the anionic dispersants gave a lower viscosity indicating better dispersion. Darven No. 2 dispersant was selected for further evaluation.

Table XVI shows results of increasing the carbon content of the Darco KB-bentonite system and the effect on viscosities. Experiment 143 gave the maximum loading at 25.4% carbon with the lowest viscosity. This was accomplished with the Darvan No. 2 as the dispersant.

Experiment 136 shows the two step procedure used to arrive at the maximum carbon loading.

With the Darco KB carbon, bentonite was selected as the thixotrope and Darvan No. 2 as the dispersant. To determine the optimum percent concentration of dispersant, a series of experiments were made and the results shown in Table XVII and Figure 4. Unlike the coal based carbon (CWS) suspensions, Darco KB wood based carbon slurries show a sharp drop in viscosity as the percent dispersant is raised to 5%. As shown in Figure 4, the viscosity of the 26% carbon loaded slurry was stabilized between 3% and 5% with only a slight decrease in viscosity at the 5% level.

The data indicates the optimum to be 3% of Darvan No. 2 dispersant with the carbon loading of 25.2%.

Table XVIII gives results of investigating sodium chromate and sodium silicate as the corrosion inhibitor. Although the suspension containing sodium silicate remained very stable during storage, the pH was unacceptably low at 6.1. Sodium chromate was selected as the corrosion inhibitor.

In finalizing the selection of the Darco KB carbon suspension, Experiments 159, 189 and 199, shown in Table XIX, were conducted. After 90 minutes mixing time, the suspensions showed large increases in viscosity with some friction heating being experienced with Experiment No. 199. Again solvent evaporation was not the apparent cause of the viscosity increase. Therefore, it is concluded that a thirty (30) minute mix time produces a uniform suspension with no improvements in stability by additional mixing.

Pigment evaluation shown by Experiments 159, 206, 207, and 208 in Table XX for the most part produced the same results as with the CWS carbon. Mapico 1100 yellow, iron oxide, gave a suitable olive green at 5.05% while higher concentrations (5.44%) of the cadmium lithopones resulted in color gradients ranging from dark green to black. Again the iron oxide pigment was chosen to give the olive green.

Of the different based carbons formulated and tested, the Darco KB appears to be the least attractive for a spray application. In order to prepare a sprayable slurry it was necessary to adjust the solvent content upward to 67.26% which gave a carbon content of 24.29%. The resulting viscosity measured 24 hours after mixing was 17,000 centipoise. As is characteristic of this particular carbon, the viscosity continues to drop during storage however does appear to stabilize after 30 days.

After formulation adjustments to give a sprayable system a typical formulation used for both testing sprayability and adhesion would be as follows:

<u>Ingredient</u>	<u>Actual Weight</u>	<u>Percent Formula Wt.</u>
Carbon: Darco KB	325 g	24.29
Thixotrope: Bentonite	6 g	.45
Dispersant: Darvan No. 2	36 g	2.69
Pigment: 1100 Yellow	65 g	4.86
Corrosion Inhibitor: Sodium Chromate	6 g	.45
Solvent: (60/40 Water-Methanol)	900 ml	67.26
Mixing Time: Thirty (30) minutes using the L-1 Laboratory Homogenizer		
Typical Viscosity at 24 hours after mixing	17,000 cps Helipath 14,800 cps #4 @ 10 RPM 7,450 cps #4 @ 20 RPM	
pH Value	10.4	
Carbon Particle Size	1-2 $\mu$ (avg.)	

TABLE XIV

## DARCO KB CARBON - THIXOTROPE EVALUATION

FORMULATION NO.	95	96	97	103	104	106		
COMPOSITION BY/WT.			Actual Wt.					
Carbon DARCO KB - Wood Based Carbon	300 g	300 g	300 g	300 g	300 g	300 g		
Thixotrope (1) Bentonite	6 g	6 g	6 g					
(2) RG-244				6 g				
(3) COK -84					6 g			
(4) Nylon-6						6 g		
Dispersant (1) DAXAD 11	12 g	12 g	12 g	12 g	12 g	12 g		
(2) Darvan No. 2								
(3) Marasperse CB								
(4) Nopcosant K								
Pigment 1100 Yellow	60 g	60 g	60 g	60 g	60 g	60 g		
Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g	6 g	6 g	6 g	6 g	6 g		
(2)								
Solvent, Water/Alcohol 60/40 By Vol.	756 ML	956 ML	856 ML	856 ML	856 ML	856 ML		
MIXING TECHNIQUE: Homogenizer, min.	--	30	30	--	30	30		

## CHARACTERISTICS

## Viscosity, CP

Imed.	Helipath "C"		10,000	16,000		14,000	12,800	
After	#4 @ 10 RPM		8,500	14,700		12,500	11,800	
Mix	#4 @ 20 RPM	2 min.	4,200	7,650	2 min.	6,300	6,050	
After	Helipath "C"		8,000	15,000		11,000	10,000	
24	#4 @ 10 RPM		7,000	13,200		10,800	9,300	
Hours	#4 @ 20 RPM		3,700	7,000		5,500	4,075	
After	Helipath "C"		7,450	13,000		9,000	9,000	
7	#4 @ 10 RPM	discarded	6,300	11,700	discarded	8,900	7,700	
Days	#4 @ 20 RPM	too viscous to mix after 2 min.	3,300	6,175	too viscous to mix after 2 min.	4,600	3,950	
After	Helipath "C"		7,000	13,000		9,000	8,200	
14	#4 @ 10 RPM		6,200	11,800		8,800	7,625	
Days	#4 @ 20 RPM		3,200	6,300		4,650	3,925	
After	Helipath "C"		8,000	14,800		11,000	8,500	
30	#4 @ 10 RPM		6,800	13,550		10,350	7,625	
Days	#4 @ 20 RPM		3,625	7,200		5,350	4,000	
pH			8.8	8.8		8.3	7.7	

NOTES:

TABLE XV

## DARCO KB CARBON - DISPERSANT EVALUATION

FORMULATION NO.	97	112	113	105		148		
COMPOSITION BY/WT.					Actual Wt.			
Carbon DARCO KB - Wood Based Carbon	300 g	300 g	300 g	300 g		300 g		
Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g		6 g		
(2)								
(3)								
(4)						(Ammonyx 856)*		
Dispersant (1) DAXAD 11	12 g					12 g		
(2) Nopcosant K		12 g						
(3) Marasperse CB			12 g					
(4) Darvan No. 2				12 g				
Pigment 1100 Yellow	60 g	60 g	60 g	60 g		60 g		
Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g	6 g	6 g	6 g		6 g		
(2)								
Solvent, Water/Alcohol 60/40 By Vol.	856 ML	856 ML	856 ML	856 ML		856 ML		
MIXING TECHNIQUE: Homogenizer, min.	30	30	30	30		30		

## CHARACTERISTICS

## Viscosity, CP

Imed.	Helipath "C"	16,000	30,000	13,000	7,000		41,000	
After	#4 @ 10 RPM	14,700	26,000	11,540	6,700		12,300	
Mix	#4 @ 20 RPM	7,650	13,950	6,000	3,475		12,100	
After	Helipath "C"	15,000	19,000	11,000	4,200		32,000	
24	#4 @ 10 RPM	13,200	18,800	9,400	3,800		12,000	
Hours	#4 @ 20 RPM	7,000	9,850	4,950	2,075		11,400	
After	Helipath "C"	13,000	18,200	7,300	4,000		28,000	
7	#4 @ 10 RPM	11,700	16,000	6,300	3,100		12,000	
Days	#4 @ 20 RPM	6,175	8,425	3,350	1,700		11,750	
After	Helipath "C"	13,000	15,000	6,000	3,800		30,000	
14	#4 @ 10 RPM	11,800	14,600	4,900	3,200		13,000	
Days	#4 @ 20 RPM	6,300	7,700	2,600	1,700		12,650	
After	Helipath "C"	14,800	16,000	6,900	4,000		30,000	
30	#4 @ 10 RPM	13,550	15,500	5,600	3,600		13,800	
Days	#4 @ 20 RPM	7,200	8,150	2,950	1,950		12,600	
pH		8.8	8.3	7.9	7.8		7.6	

TABLE XVI

## DARCO KB CARBON - % DISPERSANT EVALUATION

FORMULATION NO.	133	136	143	142				
COMPOSITION BY/WT.			Actual Wt.					
Carbon DARCO KB - Wood Based Carbon	300 g	325 g	325 g	325 g				
Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g				
(2)								
(3)								
(4)								
Dispersant (1) DAXAD 11	24 g							
(2) Darvan No. 2		12 + 12	24 g					
(3) Marasperse CB				24 g				
(4)								
Pigment 1100 Yellow	60 g	60 g	60 g	60 g				
Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g	6 g	6 g	6 g				
(2)								
Solvent, Water/Alcohol 60/40 By Vol.	856 ML	856 ML	856 ML	856 ML				
MIXING TECHNIQUE: Homogenizer, min.	30	30 + 30	30	30				

## CHARACTERISTICS

Viscosity, CP		(33,000)							
Imed.	Helipath "C"	32,000	25,000	25,000	42,000				
After	#4 @ 10 RPM	31,400	24,000	22,700	34,000				
Mix	#4 @ 20 RPM	16,500	12,300	12,200	19,250				
After	Helipath "C"	32,000	21,000	21,000	35,000				
24	#4 @ 10 RPM	31,300	20,800	20,800	30,000				
Hours	#4 @ 20 RPM	16,450	10,500	11,000	17,250				
After	Helipath "C"	32,000	20,500	19,000	24,500				
7	#4 @ 10 RPM	30,600	20,100	18,800	23,600				
Days	#4 @ 20 RPM	16,050	10,300	9,950	12,400				
After	Helipath "C"	34,000	22,000	20,000	24,500				
14	#4 @ 10 RPM	33,000	23,600	19,600	24,200				
Days	#4 @ 20 RPM	17,550	12,000	10,350	12,650				
After	Helipath "C"	34,500	25,000	23,000	31,000				
30	#4 @ 10 RPM	32,200	26,600	22,600	30,200				
Days	#4 @ 20 RPM	17,100	13,700	11,900	15,900				
pH		7.7	8.0	7.9	7.9				

NOTES:

TABLE XVII

## DARCO KB CARBON - % DISPERSANT EVALUATION

FORMULATION NO.	148	155	156	158	162
COMPOSITION BY/WT.				Actual Wt.	
Carbon DARCO KB - Wood Based Carbon	300 g	300 g	300 g	300 g	300 g
Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g	6 g
(2)					
(3)					
(4)					
Dispersant (1) Darvan No. 2	12 g	24 g	36 g	48 g	60 g
(2)					
(3)					
(4)					
Pigment 1100 Yellow	60 g	60 g	60 g	60 g	60 g
Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g	6 g	6 g	6 g	6 g
(2)					
Solvent, Water/Alcohol 60/40 By Vol.	856 ML	856 ML	856 ML	856 ML	856 ML
MIXING TECHNIQUE: Homogenizer, min.	30	30	30	30	30

## CHARACTERISTICS

## Viscosity, CP

Imed.	Helipath "C"	7,000	13,500	10,000	9,000	1,000
After	#4 @ 10 RPM	6,700	13,300	8,800	5,100	525
Mix	#4 @ 20 RPM	3,475	6,800	4,500	2,625	350
After	Helipath "C"	4,200	10,500	7,000	5,500	500
24	#4 @ 10 RPM	3,800	10,800	6,500	3,300	300
Hours	#4 @ 20 RPM	2,075	5,600	3,350	1,750	250
After	Helipath "C"					
7	#4 @ 10 RPM					
Days	#4 @ 20 RPM					
After	Helipath "C"					
14	#4 @ 10 RPM					
Days	#4 @ 20 RPM					
After	Helipath "C"					
30	#4 @ 10 RPM					
Days	#4 @ 20 RPM					
pH						

NOTES:



TABLE XVII (Cont'd)

## DARCO KB CARBON - % DISPERSANT EVALUATION

FORMULATION NO.	157	143	159	160	163			
COMPOSITION BY/WT.				Actual Wt.				
Carbon DARCO KB - Wood Based Carbon	325 g	325 g	325 g	325 g	325 g			
Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g	6 g			
(2)								
(3)								
(4)								
Dispersant (1) Darvan No. 2	12 g	24 g	36 g	48 g	60 g			
(2)								
(3)								
(4)								
Pigment 1100 Yellow	60 g	60 g	60 g	60 g	60 g			
Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g	6 g	6 g	6 g	6 g			
(2)								
Solvent, Water/Alcohol 60/40 By Vol.	856 ML	856 ML	856 ML	856 ML	856 ML			
MIXING TECHNIQUE : Homogenizer, min.	30	30	30	30	30			

## CHARACTERISTICS

## Viscosity, CP

Imed.	Helipath "C"	29,000	25,000	14,000	13,500	13,000			
After	#4 @ 10 RPM	24,800	22,700	11,900	10,400	7,450			
Mix	#4 @ 20 RPM	14,800	12,200	5,925	5,325	3,925			
After	Helipath "C"	22,000	21,000	10,000	9,000	7,500			
24	#4 @ 10 RPM	21,500	20,000	8,900	6,800	4,450			
Hours	#4 @ 20 RPM	11,950	11,000	4,550	3,575	2,500			
After	Helipath "C"								
7	#4 @ 10 RPM								
Days	#4 @ 20 RPM								
After	Helipath "C"								
14	#4 @ 10 RPM								
Days	#4 @ 20 RPM								
After	Helipath "C"								
30	#4 @ 10 RPM								
Days	#4 @ 20 RPM								
pH									

NOTES:

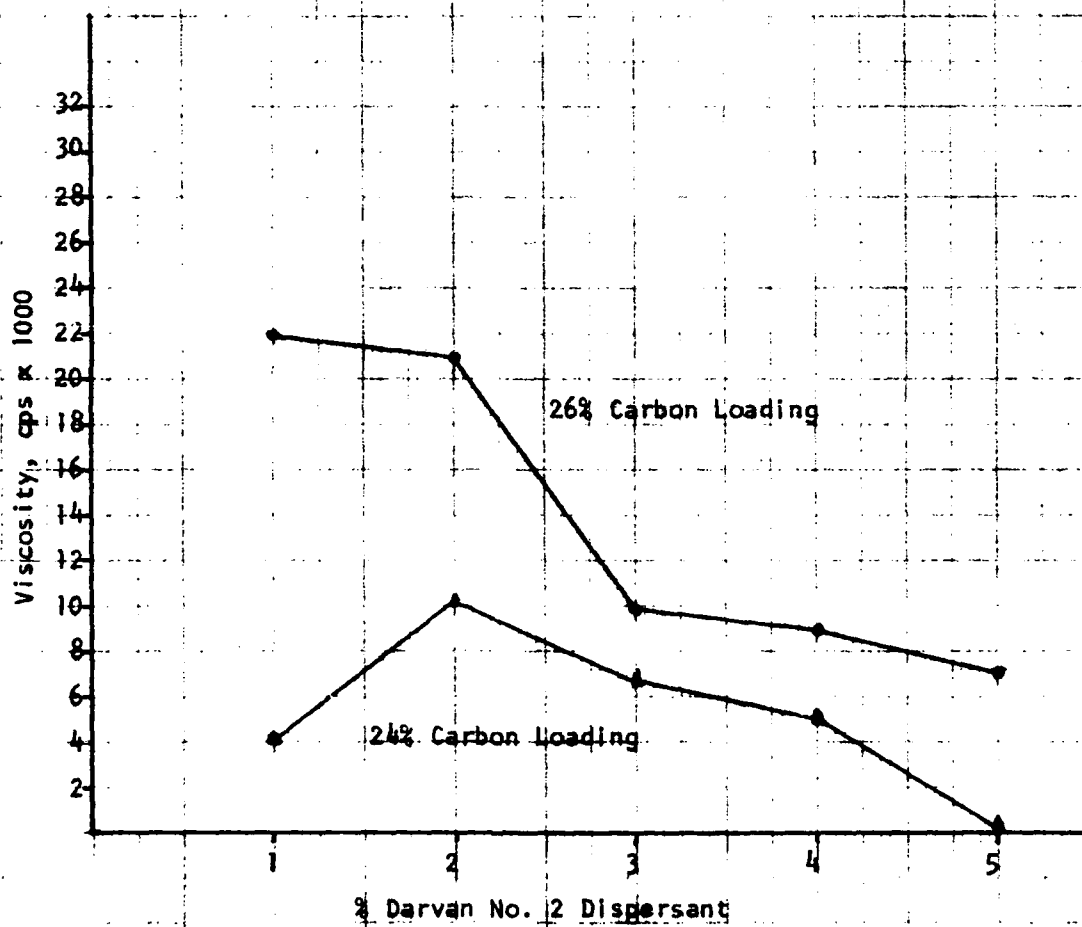


FIGURE 4 - EFFECT OF % DISPERSANT ON DARCO MB CARBON SLURRIES

TABLE XVIII

## DARCO KB - CORROSION INHIBITOR EVALUATION

FORMULATION NO.		101	102	108				
COMPOSITION BY/WT.					Actual Wt.			
Carbon	DARCO KB - Wood Based Carbon	300 g	300 g	300 g				
	Thixotrope (1) Bentonite	12 g	12 g	12 g				
	(2)							
	(3)							
	(4)							
	Dispersant (1) DAXAD 11	12 g	12 g	12 g				
	(2)							
	(3)							
	(4)							
	Pigment 1100 Yellow	60 g	60 g	60 g				
	Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g		12 g				
	(2) $\text{Na}_2\text{S}_2\text{O}_3$		6 g					
	Solvent, Water/Alcohol 60/40 By Vol.	856 ML	856 ML	856 ML				
MIXING TECHNIQUE: Homogenizer, min.		30	30	30				

## CHARACTERISTICS

## Viscosity, CP

Imed.	Helipath "C"	21,000	16,000	16,000				
After	#4 @ 10 RPM	19,200	14,500	14,350				
Mix	#4 @ 20 RPM	9,600	7,675	7,400				
After	Helipath "C"	21,000	16,000	14,000				
24	#4 @ 10 RPM	18,400	13,900	13,000				
Hours	#4 @ 20 RPM	9,200	7,450	6,800				
After	Helipath "C"	19,000	16,000	15,000				
7	#4 @ 10 RPM	16,200	13,825	13,750				
Days	#4 @ 20 RPM	8,750	7,425	7,150				
After	Helipath "C"	19,800	16,000	15,000				
14	#4 @ 10 RPM	17,050	14,200	13,640				
Days	#4 @ 20 RPM	9,200	7,650	7,075				
After	Helipath "C"	22,000	16,000	14,000				
30	#4 @ 10 RPM	19,100	15,000	13,200				
Days	#4 @ 20 RPM	11,400	7,950	6,500				
pH		8.8	6.1	8.0				

NOTES:

TABLE XIX

## DARCO KB - MIXING TIME EVALUATION

FORMULATION NO.		159	189	199	--	--		
COMPOSITION BY/WT.				Actual Wt.				
Carbon	Darco KB - Wood Based Carbon	325 g	325 g	325 g	325 g	325 g		
	Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g	6 g		
	(2)							
	(3)							
	(4)							
	Dispersant (1) Darvan No. 2	36 g	36 g	36 g	36 g	36 g		
	(2)							
	(3)							
	(4)							
Pigment	1100 Yellow	60 g	60 g	60 g	60 g	60 g		
	Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g	6 g	6 g	6 g	6 g		
	(2)							
Solvent, Water/Alcohol 60/40 By Vol. ml		856	856	856	856	856		
MIXING TECHNIQUE : Homogenizer, Minutes		30	60	90	120	180		

## CHARACTERISTICS

## Viscosity, CP

Imed.	Helipath "C"	14,000	55,000	94,000				
After	#4 @ 10 RPM	11,900	30,000	45,000				
Mix	#4 @ 20 RPM	5,925	22,000	33,000				
After	Helipath "C"	10,000	42,000					
24	#4 @ 10 RPM	8,900	27,000					
Hours	#4 @ 20 RPM	4,550	19,500					
After	Helipath "C"	8,500	42,000					
7	#4 @ 10 RPM	7,900	36,000					
Days	#4 @ 20 RPM	4,250	19,600					
After	Helipath "C"	8,000	43,000					
14	#4 @ 10 RPM	7,400	37,000					
Days	#4 @ 20 RPM	3,825	18,500					
After	Helipath "C"	8,500	44,000					
30	#4 @ 10 RPM	8,000	28,500					
Days	#4 @ 20 RPM	4,175	20,500					
In								

Solvent Content, % 59.4 58.4 --

TABLE XX

DARCO KB - PIGMENT EVALUATION

FORMULATION NO.		159	206	207	208				
COMPOSITION BY/WT.									
					Actual Wt.				
Carbon	Darco KB	325 g	325 g	325 g	325 g				
	Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g				
	(2)								
	(3)								
	(4)								
Pigment	(1) 1100 Yellow	60 g	65 g						
	(2) Azo Yellow			65 g					
	(3) Cad Yellow/Cad Orange				56/5 g				
	(4)								
Dispersant:	Darvan No. 2	36 g	36 g	36 g	36 g				
Corrosive Inhibitor (1)	$\text{Na}_2\text{C}_2\text{O}_4$	6 g	6 g	6 g	6 g				
	(2)								
Solvent, Water/Alcohol 60/40 By Vol., ml		756	756	756	756				
MIXING TECHNIQUE:	Homogenizer, minutes	30	30	30	30				

## CHARACTERISTICS

**Viscosity, CP**

	Hellipath "C"	14,000	32,000	76,000	15,000				
Imed. After Mix	#4 @ 10 RPM	11,900	27,000	After	13,500				
	#4 @ 20 RPM	5,925	14,300	2 min.	16,500				
After 24 Hours	Hellipath "C"	10,000	24,000	/	/				
	#4 @ 10 RPM	8,900	23,700	/	/				
	#4 @ 20 RPM	4,550	11,850	/	/				
	Color,	Olive	Olive		Dark				
	Wet	Green	Green	Black	Green				
	Color,	Olive	Olive		Dark				
	Dry	Green	Green	Black	Green				
pH									

NOTES:

(3) ACC Carbon - Petroleum Coke Based

The petroleum based carbon was supplied by Union Carbide in granular form. The pH value in aqueous solution measured 10.4. The surface area is 980-1080 square meters/gram with a  $\text{CCl}_4$  activity of 63%.

As discussed in the materials section of this report, a 25 pound batch of ACC carbon was ball milled for 48 hours. The average particle size after milling was  $0.7 \mu$  as determined by the Sub-Sieve Analyzer. Although this particle size is slightly less than desired (1-2  $\mu$ ), slurries were prepared for testing the thixotropic and dispersing agents. An additional 25 pound batch of ACC was prepared to the desired 1-2  $\mu$  concurrently with the experiments and was used to establish final selection of ingredients.

Experiments 98, 99, 122, 123, 124, and 125 as shown in Table XXI were prepared to test the efficiency of the bentonite, asbestos, pyrogenic silica and microcrystalline nylon as thixotropes. As the results show, bentonite was the most efficient thixotrope by giving the highest viscosities. The data also shows the effect of particle sizes on both the viscosity and stability during storage. By comparing experiments 99 and 122 it can be seen that slurries prepared with ACC carbon with an average particle size of  $1.4 \mu$  remains more stable during storage than those prepared with  $0.7 \mu$  carbon particles.

After selecting bentonite as the best thixotrope with ACC carbon, slurries were prepared to evaluate each of the dispersants. Results are shown in Table XXII with carbon loadings of 36.3%. Experiment 127 using Marasperse CB gave the lowest viscosity with ACC petroleum based carbon and was used in further evaluation.

It should be noted that results reported in Table XXII were obtained using ACC carbon with an average particle size of  $0.7 \mu$ , all of which show unacceptable stability. This series of experiments to select the most efficient dispersant was re-run using ACC carbon with a particle size of 1-2  $\mu$ .

To prepare the desired particle size, a 25 pound batch of ACC carbon was pulverized using the Mikro-Pulverizer and then ball milled for 4-1/2 hours. The average particle size after milling was  $1.10 \mu$  as determined by the Sub-Sieve Analyzer.

Using Bentonite as the thixotrope, slurries were prepared to evaluate the various dispersants with the ACC carbons. As shown in Table XXII, Nopcosant K dispersant produces an unmixable slurry, thus was eliminated from further consideration. Experiments 167, 168, and 169, shown in Table XXIII, were conducted to investigate the most efficient dispersant. Although Marasperse CB gave the lowest viscosity, Experiment 167, with a carbon loading of 36.3%, attempts to raise the viscosity by additional carbon were unsuccessful. Carbon loadings over 36.3% result in the formation of heavy glue-like texture that would be impractical to spray. Dispersants DAXAD 11 and Marasperse CB produced slurries that form the gel-like suspension after 24 hour storage.

By comparing Experiments 167 and 169, it can be seen that DAXAD 11 dispersant produces a slurry that increases in viscosity during storage, whereas the Darvan No. 2 slurry will decrease during storage. Since a decrease in viscosity during storage is more desirable for a spray application than is an increase, the Darvan No. 2 was selected as the optimum dispersant.

After selecting the Darvan No. 2 dispersing agent for use with the ACC carbon, Experiments 180, 181, 182, 183, and 186 were conducted to determine the optimum percent. As shown in Table XXIV, slurries containing 2, 3, and 4 percent of Darvan No. 2 gave no significant decreases in viscosity, however in order to produce a thixotrope capable of holding 36% carbon in suspension, it was necessary to increase the thixotrope (bentonite) content from .45% to .67%. These results are also charted in Figure 5.

Table XXV show results of evaluating sodium chromate and sodium silicate as corrosion inhibitors in the ACC carbon slurries. Although the sodium chromate resulted in a higher pH, the sodium silicate suspension gave a higher degree of stability during storage. A preliminary selection of sodium chromate was made based on the higher pH, however additional storage data proved sodium silicate to be the superior corrosion inhibitor.

The effect of additional mixing times was next evaluated on the ACC suspensions. As shown in Table XXVI, additional mixing produced the least changes in viscosity of the four different based carbons evaluated. After three (3) hours the increase was only 6,000 centipoise with approximately 4% of the solvent being lost due to evaporation. Here again the gel-like structure was formed after 24 hours storage with the thirty (30) minute mixing time. Unlike any of the other carbon suspensions tested, mixing time with the ACC suspension can be used to produce a viscosity desirable for spraying.

ACC carbon slurries containing sodium silicate as the corrosion inhibitor show a very high degree of stability. As can be seen by Table XXVII, ACC suspensions prepared with sodium silicate do not change in viscosity after static storage for 30 days and longer. Similar suspensions prepared with sodium chromate show a sharp drop in viscosity during a 30 day storage time. Although the pH is slightly lower in suspensions containing sodium silicate, (~8.0) it is felt that the high degree of stability is the more important property.

Based on the results discussed above, using the ACC carbon, the following selection can be made.

<u>Ingredient</u>	<u>Actual Weight</u>	<u>Percent Formula Wt.</u>
Carbon: ACC	470 g	34.99
Thixotrope: Bentonite	9 g	.67
Dispersant: Darvan No. 2	24 g	1.79
Pigment: 1100 yellow	78 g	5.81
Corrosion Inhibitor: Sodium Silicate	6 g	.45
Solvent: (60/40 Water-Methanol)	756 ml	55.29
Mixing Time: Thirty (30) minutes using the L-1 Laboratory Homogenizer		
Typical Viscosity at 24 hours after mixing	6,000 cps Helipath 4,000 cps #4 @ 10 RPM 2,300 cps #4 @ 20 RPM	
pH Value	8.0	
Carbon Particle Size	1.1 $\mu$ (avg.)	



TABLE XXI

## ACC CARBON - THIXOTROPE EVALUATION

FORMULATION NO.	98	99	122	123	124	125		
COMPOSITION BY/WT.	Actual Wt.							
Carbon ACC - Petroleum Coke Based	400 <sup>1</sup> g	480 <sup>2</sup> g	480 <sup>3</sup> g	480 <sup>3</sup> g	480 <sup>3</sup> g	480 <sup>3</sup> g		
Thixotrope (1) Bentonite	6 g	6 g	6 g					
(2) RG-244				6 g				
(3) COK-84					6 g			
(4) Nylon-6 (Powder)						6 g		
Dispersant (1) DAXAD 11	12 g	12 g	12 g	12 g	12 g	12 g		
(2) Darvan No. 2								
(3) Marasperse CB								
(4) Nopcosant K								
Pigment 1100 Yellow	60 g	60 g	60 g	60 g	60 g	60 g		
Corrosive Inhibitor (1) Na <sub>2</sub> C <sub>2</sub> O <sub>4</sub>	6 g	6 g	6 g	6 g	6 g	6 g		
(2)								
Solvent, Water/Alcohol 60/40 By Vol.	756 ML	756 ML	756 ML	756 ML	756 ML	756 ML		
MIXING TECHNIQUE: Homogenizer, min.	30	30	30	30	30	30		

## CHARACTERISTICS

## Viscosity, CP

Imed.	Helipath "C"	7,000	13,000	13,000	8,000	6,000	4,000		
After	#4 @ 10 RPM	5,500	11,700	8,500	4,500	4,000	2,200		
Mix.	#4 @ 20 RPM	2,800	6,100	4,650	2,450	2,150	1,250		
After	Helipath "C"	6,000	12,500	25,000	19,000	11,000	9,000		
24	#4 @ 10 RPM	5,300	11,000	18,000	16,100	6,700	6,700		
Hours	#4 @ 20 RPM	2,750	5,825	9,150	7,950	3,450	3,250		
After	Helipath "C"	6,700	12,500	23,000	19,000	10,000	8,000		
7	#4 @ 10 RPM	5,300	11,200	17,200	16,000	6,775	5,300		
Days	#4 @ 20 RPM	2,775	5,900	8,700	7,950	3,350	2,700		
After	Helipath "C"	7,000	13,000	27,000	19,000	10,000	9,000		
14	#4 @ 10 RPM	5,500	10,500	19,400	14,850	7,100	6,200		
Days	#4 @ 20 RPM	2,850	5,600	10,100	7,600	3,550	3,200		
After	Helipath "C"	7,000	12,000	34,000	30,000	16,000	13,500		
30	#4 @ 10 RPM	5,700	10,400	27,200	23,200	9,300	10,450		
Days	#4 @ 20 RPM	2,900	5,650	14,000	12,400	4,625	5,150		
pH		11.0	10.8	9.2	9.2	9.3	9.2		

## NOTES:

1. Ball milled 24 hours - Particle Size Average 1.65  $\mu$  in 1-2 lb. batch size
2. Ball milled 48 hours - Particle Size Average 1.40  $\mu$  in 1-2 lb. batch size
3. Ball milled 24 hours - Particle Size Average 0.70  $\mu$  in 25 lb. batch size

TABLE XXII

## ACC CARBONS - DISPERSANT EVALUATION

FORMULATION NO.	122	126	127	127-A	128			
COMPOSITION BY/WT.			Actual Wt.					
Carbon ACC - Petroleum Coke Based	480 g	480 g	480 g	528 g	480 g			
Thixotrope (1) Bentonite	6 g	6 g	6 g	12 g	6 g			
(2)								
(3)								
(4)								
Dispersant (1) DAXAD 11	12 g							
(2) Darvan No. 2		12 g						
(3) Marasperse CB			12 g	12 g				
(4) Nopcosant K					12 g			
Pigment 1100 Yellow	60 g	60 g	60 g	60 g	60 g			
Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g	6 g	6 g	6 g	6 g			
(2)								
Solvent, Water/Alcohol 60/40 By Vol.	756 ML	756 ML	756 ML	756 ML	756 ML			
MIXING TECHNIQUE: Homogenizer, min.	30	30	30	30	30			

## CHARACTERISTICS

## Viscosity, CP

Imed.	Helipath "C"	13,000	34,000	2,000	38,000			
After	#4 @ 10 RPM	8,500	28,800	900	19,000			
Mix.	#4 @ 20 RPM	4,650	15,300	575	10,350			
After	Helipath "C"	25,000	22,000	1,000	17,000			
24	#4 @ 10 RPM	18,000	18,350	500	7,950			
Hours	#4 @ 20 RPM	9,150	9,600	325	4,700			
After	Helipath "C"	23,000	14,000	500	7,000	Discarded - viscos to mix		
7	#4 @ 10 RPM	17,200	10,850	250	3,700			
Days	#4 @ 20 RPM	8,700	5,700	175	2,300			
After	Helipath "C"	27,000	12,000	400	6,000			
14	#4 @ 10 RPM	19,400	9,950	200	2,900			
Days	#4 @ 20 RPM	10,100	5,150	175	1,800			
After	Helipath "C"	34,000	14,500	500	7,000			
30	#4 @ 10 RPM	27,200	12,000	300	3,700			
Days	#4 @ 20 RPM	14,000	6,150	225	2,250			
pH		9.2	9.3	9.2	9.3			

## NOTES:

FORMULATION MADE WITH .7  $\mu$  CARBON

TABLE XXIII

## ACC CARBON - DISPERSANT EVALUATION

FORMULATION NO.		167	168	169				
COMPOSITION BY/WT.					Actual Wt.			
Carbon	ACC Petroleum Coke Based	480 g	480 g	480 g				
	Thixotrope (1) Bentonite	6 g	6 g	6 g				
	(2)							
	(3)							
	(4)							
	Dispersant (1) Darvan No. 2	12 g						
	(2) Marasperse CB		12 g					
	(3) DAXAD 11			12 g				
	(4) Nopcosant K	- Eliminated - (Ref. Formulation 128)						
	Pigment 1100 Yellow	60 g	60 g	60 g				
	Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g	6 g	6 g				
	(2)							
Solvent, Water/Alcohol 60/40 By Vol., ml		756	756	756				
MIXING TECHNIQUE: Homogenizer, minutes		30	30	30				

## CHARACTERISTICS

## Viscosity, CP

Imed.	Helipath "C"	13,000	1,500	10,000				
After	#4 @ 10 RPM	8,000	700	7,400				
Mix	#4 @ 20 RPM	4,350	475	4,000				
After	Helipath "C"	9,000	1,000	12,000				
24	#4 @ 10 RPM	5,600	500	10,450				
Hours	#4 @ 20 RPM	3,100	350	5,450				
After	Helipath "C"	4,900	400	13,500				
7	#4 @ 10 RPM	2,950	300	10,300				
Days	#4 @ 20 RPM	1,750	225	5,350				
After	Helipath "C"	4,000	300	15,000				
14	#4 @ 10 RPM	2,450	250	11,500				
Days	#4 @ 20 RPM	1,500	200	5,900				
After	Helipath "C"	6,000		15,000				
30	#4 @ 10 RPM	2,850		14,600				
Days	#4 @ 20 RPM	1,700		7,400				
pH		9.1	9.3	9.2				

Notes: Formulations prepared with 1.10  $\mu$  carbon.

TABLE XXIV

## ACC CARBON - PERCENT DISPERSANT EVALUATION

FORMULATION NO.	180	181	182	183		186	
COMPOSITION BY/WT.	Actual Wt.						
Carbon ACC - Petroleum Coke Based	480 g	480 g	480 g	480 g		480 g	
Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g		9 g	
(2)							
(3)							
(4)							
Dispersant (1) Darvan No. 2	12 g	24 g	36 g	48 g		24 g	
(2)							
(3)							
(4)							
Pigment 1100 Yellow	60 g	60 g	60 g	60 g		60 g	
Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g	6 g	6 g	6 g		6 g	
(2)							
Solvent, Water/Alcohol 60/40 By Vol., ml	756	756	756	756		756	
MIXING TECHNIQUE: Homogenizer, minutes	30	30	30	30		30	

## CHARACTERISTICS

## Viscosity, CP

Imed.	Helipath "C"	14,000	1,800	1,500	2,000		9,000	
After	#4 @ 10 RPM	8,150	900	900	1,225		5,250	
Mix	#4 @ 20 RPM	4,650	650	625	825		3,225	
After	Helipath "C"	7,200	700	800	1,000		4,000	
24	#4 @ 10 RPM	4,400	500	500	625		2,300	
Hours	#4 @ 20 RPM	2,650	375	375	475		1,500	
After	Helipath "C"							
7	#4 @ 10 RPM							
Days	#4 @ 20 RPM							
After	Helipath "C"							
14	#4 @ 10 RPM							
Days	#4 @ 20 RPM							
After	Helipath "C"							
30	#4 @ 10 RPM							
Days	#4 @ 20 RPM							
pH								

NOTES: Formulations prepared with 1.10  $\mu$  carbon.

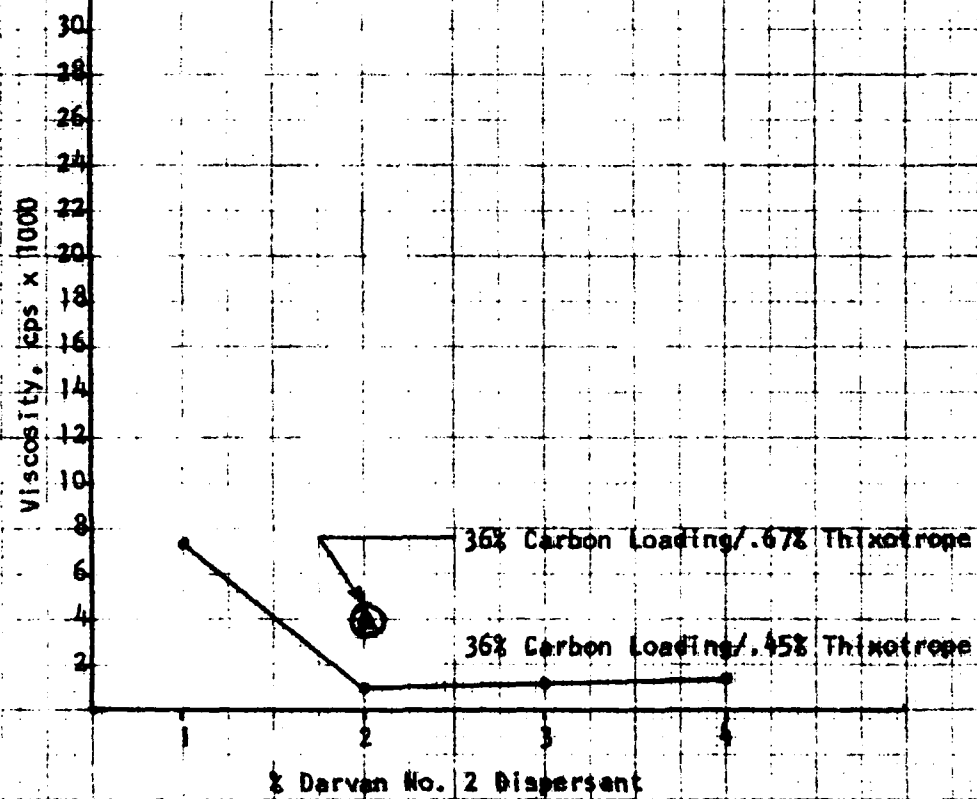


FIGURE 5 - EFFECT OF % DISPERSANT ON ACC CARBON SLURRIES

TABLE XXV

## ACC CARBON - CORROSION INHIBITOR EVALUATION

FORMULATION NO.	186	187						
COMPOSITION BY/WT.			Actual Wt.					
Carbon ACC - Petroleum Coke Based	480 g	480 g						
Thixotrope (1) Bentonite	9 g	9 g						
(2)								
(3)								
(4)								
Dispersant (1) Darvan No. 2	24 g	24 g						
(2)								
(3)								
(4)								
Pigment 1100 Yellow	60 g	60 g						
Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g							
(2) $\text{Na}_2\text{S}_2\text{O}_3$		6 g						
Solvent, Water/Alcohol 60/40 By Vol., ml	756	756						
MIXING TECHNIQUE : Homogenizer, minutes	30	30						

## CHARACTERISTICS

## Viscosity, CP

Imed.	Helipath "C"	9,000	6,000					
After	#4 @ 10 RPM	5,250	3,900					
Mix	#4 @ 20 RPM	3,225	2,400					
After	Helipath "C"	4,000	5,500					
24	#4 @ 10 RPM	2,300	3,650					
Hours	#4 @ 20 RPM	1,500	2,300					
After	Helipath "C"	1,500	6,000					
7	#4 @ 10 RPM	1,100	4,500					
Days	#4 @ 20 RPM	850	2,750					
After	Helipath "C"	1,000	6,000					
14	#4 @ 10 RPM	850	4,400					
Days	#4 @ 20 RPM	700	2,725					
After	Helipath "C"	1,000	6,000					
30	#4 @ 10 RPM	800	4,500					
Days	#4 @ 20 RPM	700	2,800					
pH		8.9	8.0					

NOTES: Particle Size  $1.10 \mu$

TABLE XXVI

## ACC CARBON - MIXING TIME EVALUATION

FORMULATION NO.	186	190	198	200	202		
COMPOSITION BY/WT.				Actual Wt.			
Carbon ACC - Petroleum Coke Based	480 g	480 g	480 g	480 g	480 g		
Thixotrope (1) Bentonite	9 g	9 g	9 g	9 g	9 g		
(2)							
(3)							
(4)							
Dispersant (1) Darvan No. 2	24 g	24 g	24 g	24 g	24 g		
(2)							
(3)							
(4)							
Pigment 1100 Yellow	60 g	60 g	60 g	60 g	60 g		
Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g	6 g	6 g	6 g	6 g		
(2)							
Solvent, Water/Alcohol 60/40 By Vol., ml	756	756	756	756	756		
MIXING TECHNIQUE: Homogenizer, minutes	30	60	90	120	180		

## CHARACTERISTICS

## Viscosity, CP

Imed.	Helipath "C"	9,000	8,000	10,500	11,000	16,000		
After	#4 @ 10 RPM	5,250	5,250	6,600	7,100	8,800		
Mix	#4 @ 20 RPM	3,225	3,200	4,000	4,350	5,300		
After	Helipath "C"	4,000	4,500	7,000	6,000	10,000		
24	#4 @ 10 RPM	2,300	2,700	4,200	3,400	6,100		
Hours	#4 @ 20 RPM	1,500	1,800	2,700	2,300	3,900		
After	Helipath "C"	1,500	2,000	3,800	5,000	7,000		
7	#4 @ 10 RPM	1,100	1,500	2,350	3,350	4,150		
Days	#4 @ 20 RPM	850	1,100	1,700	2,400	2,775		
After	Helipath "C"	1,000	1,500	2,500	4,000	5,000		
14	#4 @ 10 RPM	850	1,100	1,900	2,800	3,500		
Days	#4 @ 20 RPM	700	900	1,450	2,075	2,550		
After	Helipath "C"	1,000	1,500	3,000	6,000	6,000		
30	#4 @ 10 RPM	800	1,300	2,100	3,700	3,900		
Days	#4 @ 20 RPM	700	1,050	1,625	2,700	2,800		
pH		8.9	9.0	9.0	9.0	9.0		

NOTES: Solvent Content, % 55.7 51.8

Particle Size: 1.10  $\mu$

TABLE XXVII

## ACC CARBON - CORROSION INHIBITOR EVALUATION

FORMULATION NO.	186	190		187	211	217		
COMPOSITION BY/WT.				Actual Wt.				
Carbon ACC - Petroleum Coke Based	480 g	480 g		470 g	470 g	380 g		
Thixotrope (1) Bentonite	9 g	9 g		9 g	9 g	9 g		
(2)								
(3)								
(4)								
Dispersant (1) Darvan No. 2	24 g	24 g		24 g	24 g	24 g		
(2)								
(3)								
(4)								
Pigment 1100 Yellow	60 g	60 g		68 g	68 g	68 g		
Corrosive Inhibitor (1) $\text{Na}_2\text{Cr}_2\text{O}_4$	6 g	6 g						
(2) $\text{Na}_2\text{S}_2\text{O}_3$				6 g	6 g	6 g		
Solvent, Water/Alcohol 60/40 By Vol.	756	756		756	756	756		
MIXING TECHNIQUE: Homogenizer, Minutes	30	60		30	30			

## CHARACTERISTICS

## Viscosity, CP

Imed.	Helipath "C"	9,000	8,000		6,000	6,000	2,500		
After	#4 @ 10 RPM	5,250	5,250		3,900	4,000	1,425		
Mix	#4 @ 20 RPM	3,225	3,200		2,400	2,350	850		
After	Helipath "C"	4,000	4,500		6,000	6,000	2,500		
24	#4 @ 10 RPM	2,300	2,700		4,000	3,900	1,400		
Hours	#4 @ 20 RPM	1,500	1,800		2,300	2,300	850		
After	Helipath "C"	1,500	2,000		6,000	6,000	1,900		
7	#4 @ 10 RPM	1,100	1,500		4,500	3,850	1,000		
Days	#4 @ 20 RPM	850	1,100		2,750	2,300	600		
After	Helipath "C"	1,000	1,500		6,000	5,500	1,900		
14	#4 @ 10 RPM	850	1,100		4,400	3,700	1,000		
Days	#4 @ 20 RPM	700	900		2,725	2,300	650		
After	Helipath "C"	1,000	1,500		6,000				
30	#4 @ 10 RPM	800	1,300		4,500				
Days	#4 @ 20 RPM	700	1,050		2,800				
pH		8.9	9.0		8.0	8.0	8.0		

NOTES:

Particle Size: 1.10  $\mu$



(4) UU Carbon - Coconut Shell Based

The coconut shell based carbon used for this evaluation was supplied by Barnebey-Cheney Company in 50 x 200 mesh size particles. The pH value measured 10.4 in aqueous solution. The surface area is 14-16,000 square meters/gram with a  $\text{CCL}_4$  activity of 100%.

In order to perform the required number of experiments, a 50 pound batch of UU was ball milled for 16 hours. The average particle size as measured by the Sub-Sieve Analyzer was 0.7  $\mu$ . This carbon was used to prepare suspension for testing bentonite, asbestos, pyrogenic silica and nylon thixotropic agents. As the data in Table XXVIII shows, bentonite gave the highest viscosities and is considered to be the most efficient. In considering particle size and their effect on viscosities, experiments 100 and 129 show that it is possible to attain a much higher carbon loading with 1.2  $\mu$  than 0.7  $\mu$ . Also, the viscosity of the slurry containing the 1.2  $\mu$  is lower and more stable than the one containing the 0.7  $\mu$  carbon. Additional UU carbon was milled to the desired 1-2  $\mu$  particle size and used in making final selection of materials.

In evaluating the coconut shell based carbon with each of the candidate thixotropic agents, formulations were prepared as shown in Table XXVIII. As can be seen by comparing the five experiments, the bentonite thickening agent proved superior to the others tested.

The stability of all the thixotropes did not vary appreciably as can be seen by Experiments 100, 130, 131 and 132. Also as shown by the viscosity reading taken on Formulation Number 129, carbon particle size has a major effect on the stability as measured by viscosity.

In considering the primary objective of obtaining the highest viscosity with the least amount of thixotropic agent, the bentonite appears to be superior to the others tested.

With bentonite being the most efficient thixotrope with the UU carbon, suspensions were prepared to evaluate each of the dispersants. Experiments 129, 138, 139, and 140 shown in Table XXIX give the results of this evaluation. Using a carbon loading of 32.2% (particle size  $0.7\ \mu$ ) in each experiment, Marasperse CB appeared to give a better dispersion of carbon particles indicated by the low viscosity. Considering the effect of particle size on percent carbon loading, viscosity and stability, this series of experiments performed to select the most efficient dispersant was repeated using UU carbon with a particle size in the 1-2  $\mu$  range.

A 25 pound batch of UU carbon was pulverized using the Mikro-Pulverizer and then ball milled for 4-1/2 hours. The average particle size was measured by the Sub-Sieve Analyzer with  $1.50\ \mu$ . Having selected bentonite as the most efficient thixotrope, suspensions were prepared to evaluate the dispersants. Here again as Table XXX shows, the Nopcosant K dispersant gave unsatisfactory dispersions and was eliminated from further study. Experiment 161, 164, 165, 166, 170 and 171, listed in Table XXIX show Darvan No. 2 to be the most efficient dispersant. Using a carbon loading of 31% (particle size  $1.50\ \mu$ ) Darvan No. 2 appeared to give the best dispersion with the lowest viscosity.

Experiments 166, 170 and 171 using a concentration of 2% Darvan No. 2, show that a maximum carbon loading of 34.8% is possible for this particular dispersant.

After selecting Darvan No. 2 as the dispersing agent with UU carbon, the optimum percentage of this dispersant was next evaluated and is shown in Table XXXI. As this series of experiments show, additional amounts of dispersant over 2% had very little effect on viscosities. These results are also shown in Figure 6.

Experiments shown in Table XXXII were conducted to evaluate sodium chromate and sodium silicate as corrosion inhibitors. By comparing Experiments 170 and 179 it can be seen that suspensions prepared with sodium silicate had much higher viscosities than did the slurries containing sodium chromate with only slight increase in pH value. Sodium chromate was selected as the corrosion inhibitor.

The effect of mixing time was next evaluated using a 2% concentration of Darvan No. 2 dispersant. Results listed in Table XXXIII show that increases in mixing time give corresponding increase in viscosity. It appears that a

mix time of sixty (60) minutes will be sufficient to provide a uniform slurry that will form a gel-like structure after 24 hours and will hold the 34.8% carbon in suspension.

Table XXXIV show results of evaluating various pigments to produce an olive green. Concentrations up to ~ 5% (4.95%) of the Mapico 1100 yellow gave the desired olive green.

Although suspensions containing 34.68% UU carbon have been prepared and are sprayable, stabilization of viscosity during storage remains a problem. Unlike the CWS, where the viscosity tends to raise during storage, the UU suspensions show a sharp decrease during the first 24 hours after mix and continues to drop during storage. Although the thixotropic gel will hold the carbon in suspension and is stable from that standpoint, attempts to stabilize the viscosity over 30 days and longer storage were unsuccessful.

The optimum formulation for the UU coconut shell based carbon is made as follows:

<u>Ingredient</u>	<u>Actual Weight</u>	<u>Percent Formula Wt.</u>
Carbon: UU	455 g	34.68
Thixotrope: Bentonite	6 g	.46
Dispersant: Darvan No. 2	24 g	1.83
Pigment: 1100 Yellow	65 g	4.95
Corrosion Inhibitor: Sodium Chromate	6 g	.46
Mix Time: Sixty (60) minutes using the L-1 Laboratory Homogenizer		
Typical Viscosity at 24 hours after mixing	8,000 cps Helipath 6,000 cps #4 @ 10 RPM 3,450 cps #4 @ 20 RPM	
pH Value	9.6	
Carbon Particle Size	1.5 $\mu$ (avg.)	

TABLE XXVIII

## UU CARBON - THIXOTROPE EVALUATION

FORMULATION NO.	100	129	130	131	132			
COMPOSITION BY/WT.	Actual Wt.							
Carbon UU - Coconut Shell Based	400 <sup>1</sup> g	350 <sup>2</sup> g	350 <sup>2</sup> g	350 <sup>2</sup> g	350 <sup>2</sup> g			
Thixotrope (1) Bentonite	6 g	6 g						
(2) RG-244			6 g					
(3) COK-84				6 g				
(4) Nylon-6					6 g			
Dispersant (1) DAXAD 11	12 g	12 g	12 g	12 g	12 g			
(2) Darvan No. 2								
(3) Marasperse CB								
(4) Nopcosant K								
Pigment 1100 Yellow	60 g	60 g	60 g	60 g	60 g			
Corrosive Inhibitor (1) Na <sub>2</sub> C <sub>2</sub> O <sub>4</sub>	6 g	6 g	6 g	6 g	6 g			
(2) Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>								
Solvent, Water/Alcohol 60/40 By Vol.	756 ML	756 ML	756 ML	756 ML	756 ML			
MIXING TECHNIQUE: Homogenizer, min.	30	30	30	30	30			

## CHARACTERISTICS

## Viscosity, CP

Imed.	Helipath "C"	17,000	18,000	15,200	13,000	11,000			
After	#4 @ 10 RPM	13,500	16,200	14,800	12,900	10,800			
Mix.	#4 @ 20 RPM	6,900	8,400	7,900	6,450	5,525			
After	Helipath "C"	15,000	21,500	19,000	16,000	13,700			
24	#4 @ 10 RPM	10,800	19,300	17,800	15,500	13,000			
Hours	#4 @ 20 RPM	5,650	9,350	9,400	7,800	6,800			
After	Helipath "C"	18,000	28,000	18,800	21,000	14,000			
7	#4 @ 10 RPM	14,400	22,400	17,550	18,300	13,800			
Days	#4 @ 20 RPM	7,400	11,700	9,350	9,450	7,225			
After	Helipath "C"	20,000	33,000	18,000	21,000	13,000			
14	#4 @ 10 RPM	15,000	25,400	16,900	16,000	12,900			
Days	#4 @ 20 RPM	7,775	13,250	8,950	8,800	6,750			
After	Helipath "C"	22,000	41,000	17,000	23,000	13,000			
30	#4 @ 10 RPM	18,400	28,400	16,500	14,100	12,700			
Days	#4 @ 20 RPM	9,500	15,200	8,900	9,000	6,550			
pH		10.2	9.4	9.6	9.6	9.6			

## NOTES:

1. Ball mill 64 hours - Particle Size Average 1.2  $\mu$  - 1-2 lb batch size
2. Ball mill 16 hours - Particle Size Average 0.7  $\mu$  - 50 lb batch size

TABLE XXIX

## UU CARBON - DISPERSANT EVALUATION

FORMULATION NO.	129	138	139	140				
COMPOSITION BY/WT.			Actual Wt.					
Carbon UU - Coconut Shell Based	350 g	350 g	350 g	350 g				
Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g				
(2)								
(3)								
(4)								
Dispersant (1) DAXAD 11	12 g							
(2) Marasperse CB		12 g						
(3) Darvan No. 2			12 g					
(4) Nopcosant K				12 g				
Pigment 1100 Yellow	60 g	60 g	60 g	60 g				
Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g	6 g	6 g	6 g				
(2)								
Solvent, Water/Alcohol 60/40 By Vol.	756 ML	756 ML	756 ML	756 ML				
MIXING TECHNIQUE: Homogenizer, min.	30	30	30	30				

## CHARACTERISTICS

## Viscosity, CP

Imed.	Helipath "C"	18,000	4,000	8,700	35,000			
After	#4 @ 10 RPM	16,200	2,800	7,300	29,000			
Mix	#4 @ 20 RPM	8,400	1,500	3,900	14,900			
After	Helipath "C"	21,500	3,500	6,000	29,000			
24	#4 @ 10 RPM	19,300	1,800	4,700	19,700			
Hours	#4 @ 20 RPM	9,350	1,000	2,425	12,900			
After	Helipath "C"	28,000	2,500	4,000	21,500			
7	#4 @ 10 RPM	22,400	1,500	3,000	18,000			
Days	#4 @ 20 RPM	11,700	850	1,600	10,600			
After	Helipath "C"	33,000	3,400	4,000	19,000			
14	#4 @ 10 RPM	25,400	1,600	2,700	16,500			
Days	#4 @ 20 RPM	13,250	950	1,475	9,500			
After	Helipath "C"	41,000	5,000	4,900	17,000			
30	#4 @ 10 RPM	28,400	2,000	2,700	16,400			
Days	#4 @ 20 RPM	15,200	1,150	1,500	8,950			
pH		9.4	9.4	9.5	9.8			

NOTES:

PARTICLE SIZE 0.7  $\mu$

TABLE XXX

## UU CARBON - DISPERSANT EVALUATION

FORMULATION NO.	161	164	165		166	170	171
COMPOSITION BY/WT.	Actual Wt.						
Carbon UU - Coconut Shell Based	385 g	385 g	385 g		400 g	455 g	455 g
Thixotrope (1) Bentonite	6 g	6 g	6 g	Eliminated - (Ref. Formulation 140)	6 g	6 g	6 g
(2)							
(3)							
(4)							
Dispersant (1) DAXAD 11	12 g			- E -			
(2) Darvan No. 2		12 g			24 g	24 g	
(3) Marasperse CB			12 g				24 g
(4) Nopcosant K							
Pigment 1100 Yellow	60 g	60 g	60 g		60 g	60 g	60 g
Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g	6 g	6 g		6 g	6 g	6 g
(2)							
Solvent, Water/Alcohol 60/40 By Vol., ml	756	756	756		756	756	756
MIXING TECHNIQUE: Homogenizer, minutes	30	30	30		30	30	30

## CHARACTERISTICS

## Viscosity, CP

Imed.	Helipath "C"	12,000	8,500	9,000		6,000	13,000	19,000
After	#4 @ 10 RPM	9,400	5,600	6,600		3,825	9,400	14,300
Mix	#4 @ 20 RPM	4,800	2,950	3,400		2,075	5,150	7,400
After	Helipath "C"	14,000	4,000	7,000		2,300	3,500	5,000
24	#4 @ 10 RPM	9,900	2,600	4,500		1,450	1,900	2,600
Hours	#4 @ 20 RPM	5,175	1,475	2,400		850	1,275	1,675
After	Helipath "C"	26,000	3,700	5,000		400	1,200	2,000
7	#4 @ 10 RPM	16,200	2,100	2,200		300	850	1,300
Days	#4 @ 20 RPM	8,550	1,200	1,300		225	625	900
After	Helipath "C"	31,000	5,000	4,500			800	1,000
14	#4 @ 10 RPM	19,000	2,800	2,000			550	700
Days	#4 @ 20 RPM	10,000	1,600	1,150			475	600
After	Helipath "C"	44,000	9,000	3,500			500	600
30	#4 @ 10 RPM	22,000	4,600	1,800			450	500
Days	#4 @ 20 RPM	13,600	2,500	1,175			425	400
pH		9.6	9.6	9.6		9.5	9.5	9.7

Particle Size: 1.50  $\mu$

TABLE XXXI

## UU CARBON - PERCENT DISPERSANT EVALUATION

FORMULATION NO.	173	170	174	175			
COMPOSITION BY/WT.			Actual Wt.				
Carbon UU - Coconut Shell Based	455 g	455 g	455 g	455 g			
Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g			
(2)							
(3)							
(4)							
Dispersant (1) Darvan No. 2	12 g	24 g	36 g	48 g			
(2)							
(3)							
(4)							
Pigment 1100 Yellow	60 g	60 g	60 g	60 g			
Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g	6 g	6 g	6 g			
(2)							
Solvent, Water/Alcohol 60/40 By Vol., ml	756	756	756	756			
MIXING TECHNIQUE: Homogenizer, minutes	30	30	30	30			

## CHARACTERISTICS

## Viscosity, CP

Imed.	Helipath "C"	28,000	13,000	13,000	14,000			
After	#4 @ 10 RPM	20,000	9,400	8,600	9,250			
Mix	#4 @ 20 RPM	13,000	5,150	4,750	5,100			
After	Helipath "C"	16,000	3,500	4,000	3,900			
24	#4 @ 10 RPM	13,200	1,900	2,300	2,300			
Hours	#4 @ 20 RPM	6,800	1,275	1,450	1,425			
After	Helipath "C"							
7	#4 @ 10 RPM							
Days	#4 @ 20 RPM							
After	Helipath "C"							
14	#4 @ 10 RPM							
Days	#4 @ 20 RPM							
After	Helipath "C"							
30	#4 @ 10 RPM							
Days	#4 @ 20 RPM							
pH								

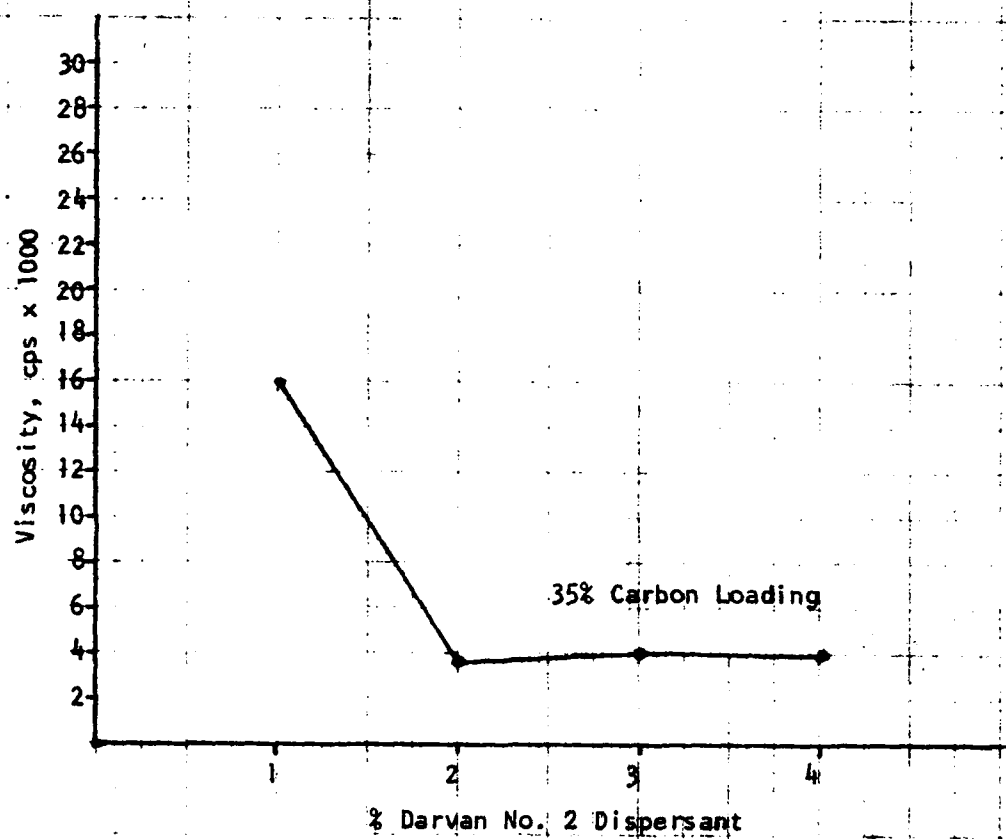


FIGURE 6 - EFFECT OF % DISPERSANT ON UU CARBON SLURRIES



TABLE XXXII

## UU CARBON - CORROSION INHIBITOR EVALUATION

FORMULATION NO.	170	179						
COMPOSITION BY/WT.			Actual Wt.					
Carbon UU - Coconut Shell Based	455 g	455 g						
Thixotrope (1) Bentonite	6 g	6 g						
(2)								
(3)								
(4)								
Dispersant (1) Darvan No. 2	24 g	24 g						
(2)								
(3)								
(4)								
Pigment 1100 Yellow	60 g	60 g						
Corrosive Inhibitor (1) $\text{Na}_2\text{CO}_3$	6 g							
(2) $\text{Na}_2\text{SiO}_3$		6 g						
Solvent, Water/Alcohol 60/40 By Vol., ml	756	756						
MIXING TECHNIQUE: Homogenizer, minutes	30	30						

## CHARACTERISTICS

## Viscosity, C

Imed.	Helipath "C"	13,000	62,000					
After	#4 @ 10 RPM	9,400	21,800					
Mix.	#4 @ 20 RPM	5,150	13,250					
After	Helipath "C"	3,500	54,000					
24	#4 @ 10 RPM	1,900	22,200					
Hours	#4 @ 20 RPM	1,275	12,000					
After	Helipath "C"	1,200						
7	#4 @ 10 RPM	850						
Days	#4 @ 20 RPM	625						
After	Helipath "C"	800						
14	#4 @ 10 RPM	550						
Days	#4 @ 20 RPM	475						
After	Helipath "C"	500						
30	#4 @ 10 RPM	450						
Days	#4 @ 20 RPM	425						
pH		9.5	9.7					

NOTES:

TABLE XXXIII

## UU CARBON - MIXING TIME EVALUATION

FORMULATION NO.	170	194	195	196	197
COMPOSITION BY/WT.				Actual Wt.	
Carbon      UU - Coconut Shell Based	455 g	455 g	455 g	455 g	455 g
Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g	6 g
(2)					
(3)					
(4)					
Dispersant (1) Darvan No. 2	24 g	24 g	24 g	24 g	24 g
(2)					
(3)					
(4)					
Pigment      1100 Yellow	60 g	60 g	60 g	60 g	60 g
Corrosive Inhibitor (1) Na <sub>2</sub> C <sub>2</sub> O <sub>4</sub>	6 g	6 g	6 g	6 g	6 g
(2)					
Solvent, Water/Alcohol 60/40 By Vol., ml	756	756	756	756	756
MIXING TECHNIQUE: Homogenizer, minutes	30	60	90	120	180

## CHARACTERISTICS

**Viscosity, CP**

Imed.	Helipath "C"	13,000	27,000	21,500	30,000	49,000			
After Mix	#4 @ 10 RPM	9,400	18,800	16,900	22,000	29,000			
	#4 @ 20 RPM	5,150	10,050	9,000	11,800	16,000			
After 24 Hours	Helipath "C"	3,500	9,000	10,000	15,500	24,000			
	#4 @ 10 RPM	1,900	6,900	7,600	11,000	19,800			
	#4 @ 20 RPM	1,275	3,975	4,300	6,200	10,800			
After 7 Days	Helipath "C"	1,200	5,000	7,000	13,000	20,500			
	#4 @ 10 RPM	850	3,500	4,700	8,000	15,100			
	#4 @ 20 RPM	625	2,250	2,950	4,725	8,400			
After 14 Days	Helipath "C"	800	4,000	6,000	10,000	23,000			
	#4 @ 10 RPM	550	3,000	4,500	7,600	17,400			
	#4 @ 20 RPM	475	1,950	2,850	4,600	9,700			
After 30 Days	Helipath "C"	500	5,000	10,000	16,000	34,000			
	#4 @ 10 RPM	450	3,550	6,300	11,500	22,700			
	#4 @ 20 RPM	425	2,350	3,900	6,750	12,400			
pH		9.5	9.6	9.6	9.6	9.6			
Solvent Content, %		58.9				57.9			

-72-

**TABLE XXXIV**

## UU CARBON - PIGMENT EVALUATION

FORMULATION NO.	170	203	204	205			
COMPOSITION BY/WT.				Actual Wt.			
Carbor. UU - Coconut Shell Based	455 g	455 g	455 g	455 g			
Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g			
(2)							
(3)							
(4)							
Pigment (1) 1100 Yellow	60 g	65 g					
(2) Cad Yellow/Cad Orange			56/5 g				
(3) Azo Yellow				65 g			
(4)							
Dispersant: Darvan No. 2	24 g	24 g	24 g	24 g			
Corrosive Inhibitor (1) $\text{Na}_2\text{C}_2\text{O}_4$	6 g	6 g	6 g	6 g			
(2)							
Solvent, Water/Alcohol 60/40 By Vol., ml	756	756	756	756			
MIXING TECHNIQUE: Homogenizer, Minutes	30	30	30	30			

## CHARACTERISTICS

**Viscosity, CP**

[illegible]

d. The Formation of Thixotropic Gels

In working with the four different based carbons, it was observed that during the first three days storage of certain carbon slurries, a thixotropic, non-pourable, uniform gel is formed. This is considered a very important and desirable condition in that it prevents the settling out of the carbon particles. It is believed that this gel forms as a result of the interaction of bentonite with the active carbon.

This gel-like structure is very shear sensitive and can be easily made fluid by one or two shakes of the container.

It can be concluded from the data presented in Table XXXV that slurries with high undisturbed viscosities as high as 102,000 centipoise (and possibly higher) can be easily reconstituted into a fluid state with lowered viscosities that can be easily sprayed. For example, Formulation No. 138 with an undisturbed viscosity of 102,000 centipoise reconstitutes easily to 6,000 centipoise.

Of equal importance is the fact that the gel-like structure will reform repeatedly after being reconstituted to the fluid state.

As required by the contract, samples of each of the optimized formulations were submitted to Edgewood Arsenal for evaluation and testing. A "B" portion of each sample was retained at Brunswick to monitor the effect of static storage. Table XXXVI shows the effect of storage up to 142 days. It is felt that, based on this data, storage for 2 years will not adversely effect these carbon loaded thixotropes.

TABLE XXXV

## FORMATION OF A THIXOTROPIC GEL

Form No. CWS Carbon	Viscosity		Amount Free Solvent	Amount Settling	Gel Formation <sup>(2)</sup> L-M-H	Color	Easily Reconsti- tuted	Notes
	Undist.	Reconsti- tution <sup>(1)</sup>						
115	70,000	16,000	20 ml	0	Yes (L)	Green	Yes	
116	--	--	100 ml	100%	No (H)	--	No	
117	136,000	8,000	82 ml	50%	Yes (M)	Green	Yes	
118	--	--	--	100%	No (H)	Green	No	
120	40,000	1,200	23 ml	0	Yes (L)	Green	Yes	
137	>200,000	3,000	6 ml	0	Yes (H)	Green	No	Very heavy glue-like
141	140,000	2,000	3 ml	0	Yes (M)	Green	Yes	
144	74,000	2,000	8 ml	0	Yes (L)	Green	Yes	
145	163,000	34,000	11 ml	0	Yes (H)	Green	Yes	Hard gel but easily reconsti- tuted
ACC Carbon <sup>(3)</sup>								
122	65,000	30,000	6 ml	0	Yes (L)	Black	Yes	
123	58,000	24,000	15 ml	0	Yes (L)	Black	Yes	
124	74,000	14,000	20 ml	0	Yes (L)	Black	Yes	
125	50,000	12,000	27 ml	0	Yes (L)	Black	Yes	
126	70,000	17,000	9 ml	0	Yes (M)	Black	Yes	

TABLE XXXV (Cont'd)

## FORMATION OF A THIXOTROPIC GEL

Form No. UU Carbon	Viscosity		Amount Free Solvent	Amount Settling	Gel Formation L-M-H(2)	Color	Easily Reconsti- tuted	Notes
	Undist.	Reconsti- tution (1)						
100	>200,000	30,000	9 ml	0	Yes (M)	Green	Yes	
129	136,000	50,000	6 ml	0	Yes (M)	Green	Yes	
130	110,000	15,000	34 ml	0	Yes (M)	Green	Yes	
131	>200,000	27,000	34 ml	0	Yes (M)	Green	Yes	
132	100,000	10,000	39 ml	0	Yes (L)	Green	Yes	
138	102,000	6,000	21 ml	0	Yes (L)	Green	Yes	
139	74,000	5,000	16 ml	0	Yes (L)	Green	Yes	
140	82,000	17,500	18 ml	0	Yes (L)	Green	Yes	
DARCO KB Carbon								
97	57,000	19,000	7 ml	0	Yes (L)	Green	Yes	
104	>200,000	11,000	62 ml	30%	No (H)	Green	Yes	
106	>200,000	15,000	44 ml	30%	No (H)	Green	No	
112	44,000	19,000	7 ml	0	Yes (L)	Green	Yes	
113	30,000	9,500	5 ml	0	Yes (L)	Green	Yes	
105	29,000	5,000	13 ml	0	Yes (L)	Green	Yes	
133	90,000	36,000	2 ml	0	Yes (L)	Green	Yes	
136	104,000	28,000	2 ml	0	Yes (L)	Green	Yes	
142	130,000	32,000	2 ml	0	Yes (L)	Green	Yes	
143	108,000	28,000	6 ml	0	Yes (L)	Green	Yes	

(1) Reconstituted, Helipath

(2) Light, Medium, Hard

(3) Suspension Prepared with .7  $\mu$  Particle Size Carbon

TABLE XXXVI

## STATIC STORAGE TEST OF CARBON THIXOTROPES

Form No.	Static Storage, Days	Viscosity		Free Solvent	Amount Settling	Gel Formation L-M-H(2)	Ease of Reconstitution
		Undist.	Reconstituted (1)				
B-CWS-1	90	180,000	27,000	16.0	0	M	Yes
B-ACC-1A	90	120,000	13,500	.5	0	L	Yes
B-ACC-1B	90	120,000	10,000	7.0	0	L	Yes
B-ACC-2	90	44,000	3,000	16.0	0	L	Yes
B-UU-1	90	200,000+	4,500	10.0	0	M	Yes
B-KB-1	90	79,000	7,500	12.0	0	L	Yes
E-ACC-1-A	142	134,000	14,000	7.0	0	M	Yes
E-ACC-2-A	142	51,000	4,000	20.0	0	L	Yes

(1) Reconstituted, Helipath

(2) Light-Medium-Hard

#### D. Spraying and Adhesion Testing

##### 1. Spray Experiments

To begin spraying experiments with each of the optimized formulations, two 24" x 24" aluminum test panels were spray coated with 1 mil of chromate primer and 1 mil of OG lusterless alkyd lacquer. Test tabs (1 in<sup>2</sup>) were coated along with the panel and were used for determining the amount of carbon deposited on the surface after each test. The standard U.S. Army sprayer unit equipped with the standard nozzle and .033" diameter atomizing hole was used to test each of the different based carbons. Figure 7 and 8 show the spray set-up and coated test panel.

##### Spray Test No. 1 - CWS Carbon

The optimized formulation as discussed in Section II-C was used for this test. The viscosity measured in the 14,000 centipoise range. The standard sprayer was filled with 1 liter and pressurized to 200 psi from a nitrogen (standard 2500 cubic feet) tank. The 24" x 24" test panel was set 90° to the sprayer nozzle at a distance of 5 feet. One pass was made over the entire panel. As the slurry tends to be broken up into very small droplets, one pass was not sufficient to cover the surface of the panel. Two additional passes were then made which covered the surface completely. After allowing the coating to dry, the test tabs were removed to determine coverage.

<u>Test Tab No.</u>	<u>Carbon Deposited</u>
1	22.4 mg/cm <sup>2</sup>
2	19.2 mg/cm <sup>2</sup>
3	21.9 mg/cm <sup>2</sup>
4	19.6 mg/cm <sup>2</sup>

Because the slurry is being dispensed in very small droplets, requiring 2-3 passes for complete surface coverage, high carbon deposits are being recorded as shown above.

##### Spray Test No. 2 - Darco KB Carbon

The same general procedure was used to test sprayability of the Darco KB suspensions. The viscosity measured in the 17,000 centipoise range. A total of 3 passes was made over the panel with the following coverage:

<u>Test Tab No.</u>	<u>Carbon Deposited</u>
1	12.6 mg/cm <sup>2</sup>
2	11.8 mg/cm <sup>2</sup>
3	10.4 mg/cm <sup>2</sup>
4	9.7 mg/cm <sup>2</sup>



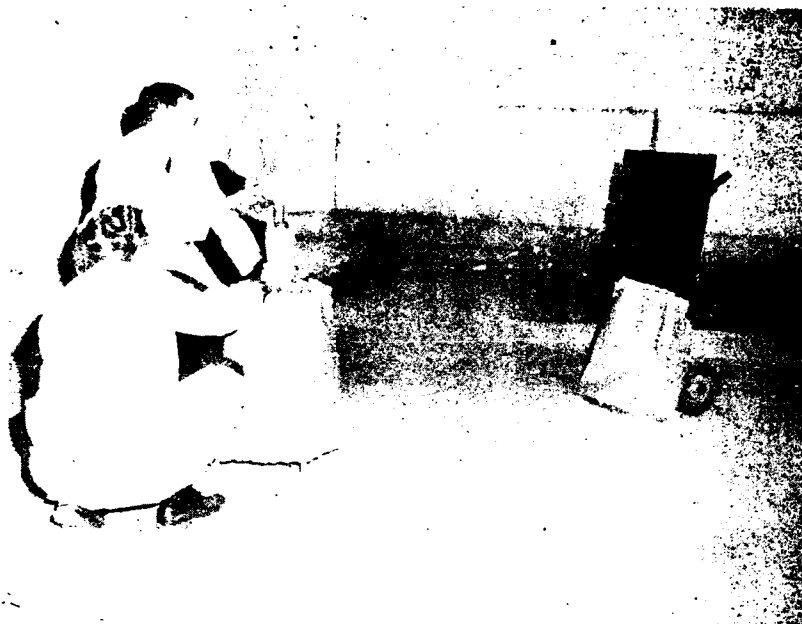


FIGURE 7 - SPRAY TESTING APPARATUS TO DETERMINE  
SPRAYABILITY AND COVERAGE

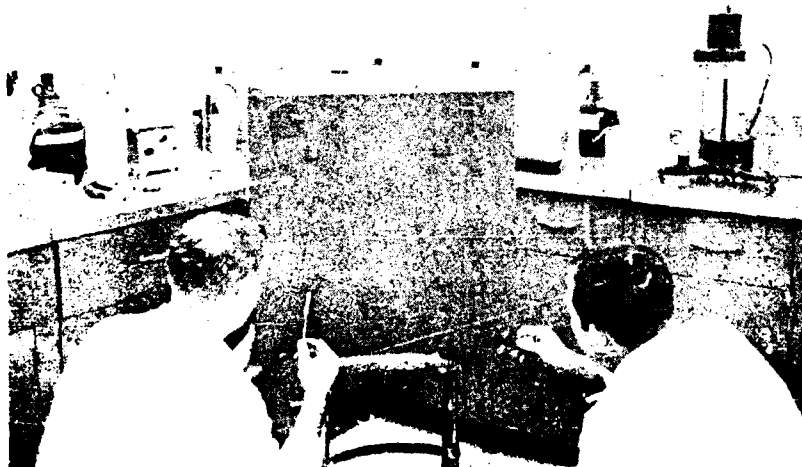


FIGURE 8 - CARBON COATED PANEL WITH TEST TABS  
TO MEASURE COVERAGE

Again the spray was in the form of very small droplets requiring multiple passes for complete surface coverage.

Spray Test No. 3 - ACC Carbon

Although the ACC suspension sprayed better than did the other carbon slurries, the surface coating was similar to that of the previous tests. Again the spray was in the form of small droplets. The viscosity measured in the 6,000 cps range. After 3 passes over the test panel the coverage measured as follows:

<u>Test Tab No.</u>	<u>Carbon Deposited</u>
1	10.6 mg/cm <sup>2</sup>
2	10.1 mg/cm <sup>2</sup>
3	10.4 mg/cm <sup>2</sup>
4	10.8 mg/cm <sup>2</sup>

The spray tests conducted on the UU suspension gave results very similar to those obtained using the CWS and Darco KB slurries. Each test tab showed an average of 12 mg/cm<sup>2</sup> carbon coating.

By considering stability and sprayability of the different carbon based thixotropes, the suspension prepared with the ACC activated carbon was chosen for additional spray tests.

Spray Test No. 4 - ACC Carbon

To further test the sprayability of the ACC suspensions, a standard DeVilbiss paint spray gun was used to prepare a test panel. The spray gun was a type MBC with AV15EX head and DEX needle and was operated at 68 psi air pressure.

From a distance of 15-18 inches, 2 passes were made over the test panel. The resulting spray, fog rather than droplets, gave a uniform even coating. The test tabs were weighed for coverage and the following results were obtained.

<u>Test Tab No.</u>	<u>Carbon Deposited</u>
1	4.56 mg/cm <sup>2</sup>
2	3.72 mg/cm <sup>2</sup>
3	4.43 mg/cm <sup>2</sup>
4	3.72 mg/cm <sup>2</sup>

For comparison, a spray test was made with the same ACC suspension but spraying with the standard apparatus. From a distance of 5 feet, operating at 200 psi nitrogen pressure, 3 passes were necessary over the test panel to cover the surface. The test tabs measured the following coverage.

<u>Test Tab No.</u>	<u>Carbon Deposited</u>
1	8.2 mg/cm <sup>2</sup>
2	7.8 mg/cm <sup>2</sup>
3	9.2 mg/cm <sup>2</sup>
4	8.1 mg/cm <sup>2</sup>

In viewing Figures 9 and 10, it can be seen that a more uniform, even coating is applied using the DeVilbiss gun than was possible with the existing standard spray unit. The panel prepared with the standard apparatus has the larger carbon deposit (8-9 mg/cm<sup>2</sup>) but has evidence of uncoated areas caused by the droplet form spray.

It can be concluded from the above tests that with some modification to the standard spray apparatus, the ACC carbon slurries can be sprayed satisfactorily to give the desired 3-4 mg/cm<sup>2</sup> activated carbon coating.

## 2. Adhesion Tests

Adhesion tests were next performed using formulations as described in Section II-C. In preparing for abrasion tests, three aluminum panels 3" x 6" were coated with 1 mil of chromate primer and 1 mil of lusterless OG alkyd lacquer. These test panels along with test tabs were then coated with the activated carbon slurry as follows:

Spray Gun:   DeVilbiss Type MBC  
                   AV15-EX Nozzle/DEX Needle

Distance:    15-18 inches panel to spray gun

Pressure:     100 pounds air pressure

Passes:       2 in 7-10 seconds

After drying for twelve hours at 72°F and 50% R.H., the test tabs were reweighed to determine coverage. Figures 11 and 12 show the test panel before and after spraying.

Terry cloth strips 3.16 x 3.16 centimeters (10 cm<sup>2</sup> surface area) were cut for abrasion pads. A 10 mil thick rigid plastic film was placed over the terry cloth to evenly distribute the 100 gram weight. The weighed terry cloth was dragged over the carbon coating at a rate of 5 inches per minute using the Instron Test Machine (see Figures 13 and 14). The percent carbon loss was calculated as follows:

$$\frac{W_2 - W_1}{P_A} = W_A \text{ (gm/cm}^2\text{)}$$

$$\frac{W_2 - W_3}{T_A} = W_B \text{ (g/cm}^2\text{)}$$

$$\frac{W_B}{W_A} \times 100 = \% \text{ (g/cm}^2\text{) Carbon Loss}$$

Where:  $W_1$  - Weight of test panel  
 $W_2$  = Weight of carbon coated test panel  
 $W_A$  = Amount of carbon coating per unit area  
 $P_A$  = Area of test panel in  $\text{cm}^2$   
 $W_3$  = Weight of test panel after abrasion  
 $T_A$  = Area of the abrasion path in  $\text{cm}^2$   
 $W_B$  = Amount of carbon abraded away

Table XXXVII shows results of the abrasion tests for each of the different based carbons. It can be seen that all carbons are well within the maximum allowable loss of 25%.



FIGURE 9 - CLOSE-UP OF ACTIVATED CARBON COATING  
 SPRAYED WITH THE STANDARD SPRAYER APPARATUS



FIGURE 10 - CLOSE-UP OF ACTIVATED CARBON COATING  
 SPRAYED WITH A DEVILBISS MBC SPRAY GUN

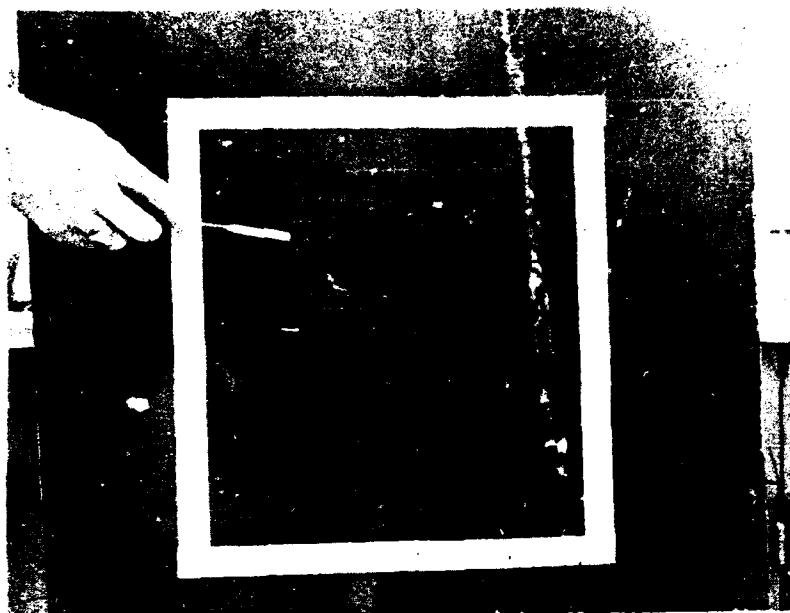


FIGURE 11 CARBON COATED PANEL WITH TEST TABS  
TO MEASURE COVERAGE



FIGURE 12 ABRASION TEST PANELS WITH A  $3-4 \text{ mg/cm}^2$   
COATING OF ACTIVATED CARBON



FIGURE 13 ABRASION TEST SET-UP  
USING INSTRON TEST MACHINE

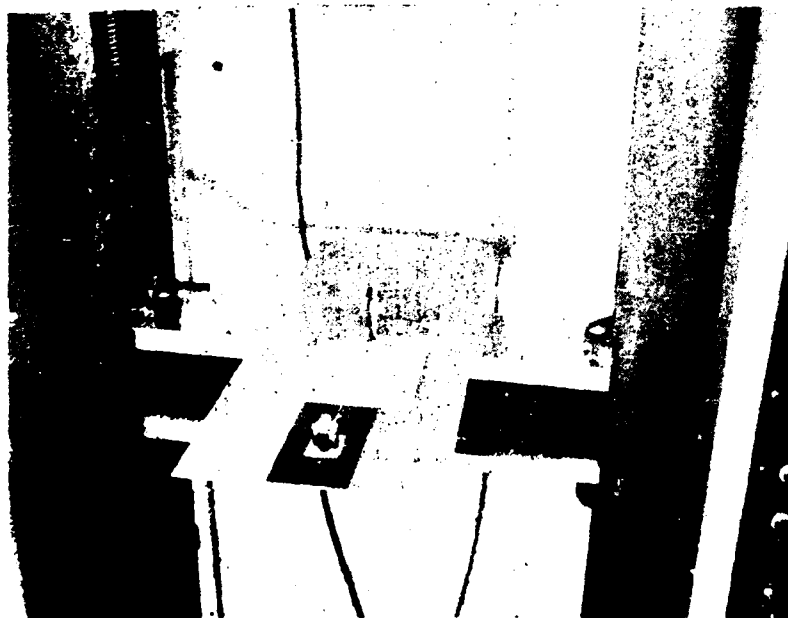


FIGURE 14 CLOSE-UP SHOWING 100 GRAM  
WEIGHT AND TERRY CLOTH

TABLE XXXVII

ABRASION RESISTANCE OF ACTIVATED CARBON  
DEPOSITED ON ALKYD LACQUER COATED SURFACES

<u>Carbon</u>	<u>Test Panel Number</u>	<u>Carbon Coating mg/cm<sup>2</sup></u>	<u>Carbon Loss Percent</u>
1. <u>CWS</u>	1	3.87	3.30
Coal Based	2	3.36	6.06
	3	3.55	2.84
2. <u>Darco KB</u>	10	5.12	4.78
Wood Based	11	5.49	4.58
	12	4.99	5.18
3. <u>ACC</u>	7	4.01	6.36
Petroleum Based	8	4.80	7.19
	9	3.83	6.71
4. <u>UU</u>	4	3.87	12.09
Coconut Shell Based	5	3.75	8.92
	6	3.27	6.74



### III. PHASE B - CLOTHING AND INDIVIDUAL EQUIPMENT DECONTAMINATION

Phase II of the program involves the formulation of dry forms of activated carbon, inert filler and pigment that will give maximum agent vapor absorption when applied onto a surface such as woven uniform fabric material.

Primary objectives are to (1) formulate a dry mixture that will give the highest possible adherence to the surface after some degree of abrasion, and (2) can be easily applied by sprinkling and/or rubbing to give a coating of 3-4 mg/cm<sup>2</sup> of dry activated carbon.

#### A. Establishment of Basic Materials, Equipment and Methods of Investigation

Work reported in this report details how selections were made of materials, equipment, and methods of investigation.

##### 1. Government Supplied Materials

The following materials were furnished by Edgewood Arsenal as a part of this contract.

- Carbon - Coal based, CWS type manufactured by Pittsburgh Activated Carbon Co. The average particle size of the carbon is 1-2 microns.
- Uniform Fabric - Five (5) yard samples of (1) 6 oz. poplin cotton, (2) 8.2 oz. sateen cotton, and (3) 9 oz. Quarpel-treated Nyco sateen uniform material.

##### 2. Inert Filler Materials

The following filler materials were considered for this application.

- Asbestos - Samples of each of the two basic types of asbestos were obtained for formulating.
  - Type I - Serpentine - Grade 4A3, supplied by Van Horn Metz Co.
  - Type II - Amphibole - Grade 25-PM, supplied by Powhatan Mining Company.
- Fibers - Other fibrous materials were also considered. These include nylon, milled glass and polyethylene.
- Pigment - To give an olive green, iron oxide pigment was used.

### 3. Mixing Techniques and Equipment

Basically, two different mixing techniques were evaluated, i.e., wet and dry. In wet mixing the basic ingredients are combined in an aqueous medium after which it is vacuum filtered and dried. The dry mixing simply involves combining the dry ingredients together by a mechanical mixer. It was the intention of the investigation to establish a method whereby the dry mixing technique will produce a formulation with the same high degree of uniformity as is obtained by wet mixing.

The equipment used in preparation of the carbon formulations was the L-1 Laboratory Homogenizer and the Bantam Mikro-Pulverizer. The pulverizer was used to mill the long fiber asbestos (Serpentine) to the desired length as well as preparation of the dry mixes.

### 4. Auxiliary Equipment

Other equipment used in the investigation of these materials included the following:

Applicator Bags - The bags were prepared from cotton knit type fabric and measure approximately 3-1/2" x 6". A nylon scrim "window" was stitched into one side of the applicator and measures 2-1/4" x 4". The "window" is woven with approximately .050" openings between strands and is approximately .010" thick. These applicator bags, along with similar ones supplied by Edgewood, were used to apply a variety of dry carbon mixes to fabric surfaces.

Abrasion Test Apparatus - An abrasion test apparatus was constructed according to the design suggested by Edgewood Arsenal. The apparatus is designed to tumble a carbon loaded woven fabric to simulate the abrading effect resulting from a uniform being worn for a twenty-four (24) hour period. This test machine is described in detail in a later section of this report.

Ball Mill - A two-gallon ball mill was used to tumble selected formulation in an effort to improve the homogeneity. Baffles were later added to the ball mill to improve the mixing effect.

## B. Formulation and Testing of Dry Carbon Mixtures

To begin the investigation of dry forms of activated carbon for an absorbent decontaminant, certain process variables were first established. These process variables include (1) preparation of an asbestos filler prior to the mixing operation, and (2) the effect on the homogeneity of the mixture when prepared in an aqueous medium or simply mixed dry.

The preparation of the filler asbestos (Grade 4A3 - Serpentine) consists of braking up the long fibers into short acicular needles with "exploded" ends to promote both adhesion to woven fabric and entrap the maximum amount of carbon particles. This is accomplished by pulverizing the asbestos through a hammer mill fitted with a fine screen. The size screens evaluated for this application ranged from .010" to .062" openings.

### 1. Microscopic Examination

Samples of the 4A3 asbestos were first prepared (pulverized) for microscopic examination. Glass slides were prepared by mixing small amounts of asbestos in methanol, placing a drop onto the slide and allowing to dry.

Figure 15 shows the long asbestos fibers as received from the Van Horn Metz Company. Figure 16 shows the asbestos after pulverizing through a .010" x .500" slotted screen. It can be seen that the individual fibers are broken up into different lengths ranging from a few microns up to several thousand microns. Figure 17 shows the results of pulverizing the asbestos through a screen having .032" diameter holes. The individual fibers are broken into more uniform lengths than are those put through the .010" x .500" slotted screen. Figure 18 is the result of a screen having .040" diameter holes. As the photograph shows, much longer fibers are getting through the .040" diameter hole than with the previous described screens. Figure 19 is asbestos milled through a screen having .062" diameter openings. As can be seen, only a small percentage of the fibers were broken up into desirable lengths. As the length fibers do not promote sufficient adhesion or entrapment of carbon particles, the .062" perforated screen was eliminated from further evaluation.

Samples of the amphibole type asbestos (shorts) were also examined through the microscope. Figure 20 is a typical example of asbestos "shorts" as received from the supplier. As can be seen, amphibole is composed of approximately 50% fibers and 50% crystalline masses of asbestos. As the fibers provide mechanical adhesion where the crystalline structures will not, it is felt that the amphibole type asbestos does not warrant additional evaluation.



FIGURE 15  
CRYSOTILE (TYPE 4A3) ASBESTOS AS RECEIVED FROM VENDOR



FIGURE 16  
4A3 ASBESTOS FIBERS AFTER MILLING THROUGH  
A .010" x .500" SLOTTED SCREEN



FIGURE 17

4A3 ASBESTOS FIBERS AFTER MILLING THROUGH  
A .032" PERFORATED SCREEN



FIGURE 18

4A3 ASBESTOS FIBERS AFTER MILLING THROUGH  
A .040" PERFORATED SCREEN

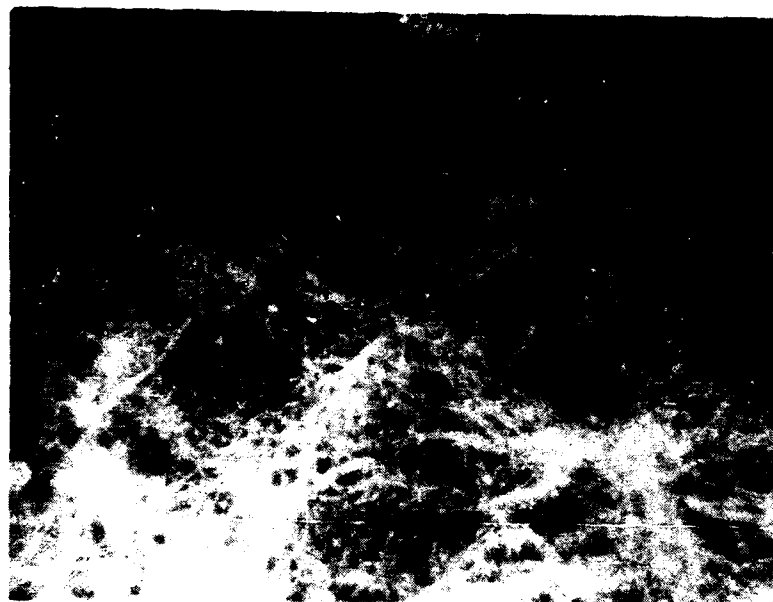


FIGURE 19

4A3 ASBESTOS FIBERS AFTER MILLING THROUGH  
A .062" PERFORATED SCREEN

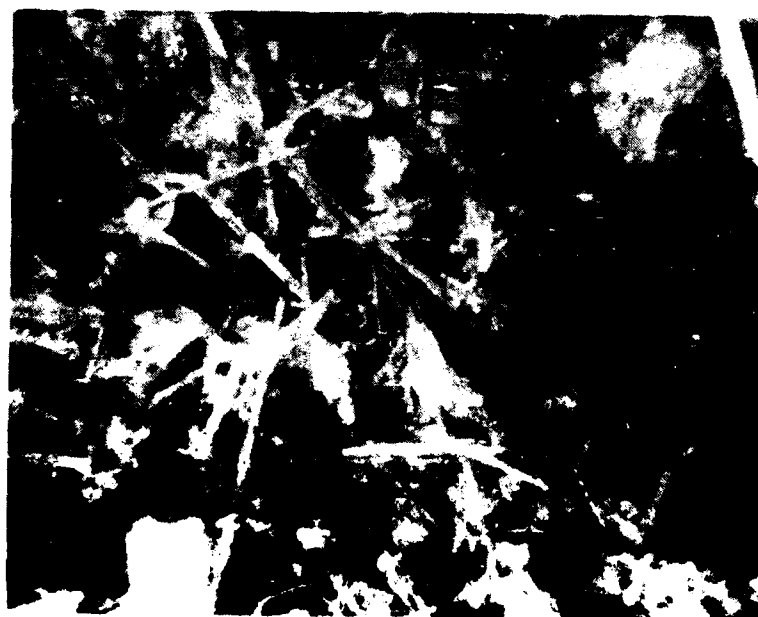


FIGURE 20

AMPHIBOLE TYPE ASBESTOS (SHORTS)  
AS RECEIVED FROM THE SUPPLIER

The two basic types of asbestos discussed above are defined as follows. Type I, Serpentine, (commonly called "crysotile") consists of long flexible fibers composed almost entirely of magnesium silicate. Type II, Amphibole, is a shorter fiber form asbestos, with more brittle fibers composed of various silicates such as magnesium, iron, calcium and sodium. The type is usually referred to as "shorts".

## 2. Preparation of Wet and Dry Formulations

In preparation of the wet mixed formulation, the general procedure was as follows:

Formulation:            1,000 ml of distilled water  
                             5 grams of 4A3 asbestos  
                             100 grams of CWS carbon  
                             10 grams of iron oxide pigment

### Operations:

The 4A3 asbestos is first milled through the Mikro-Pulverizer to reduce the fiber length. Five (5) grams were then added to the 1000 ml of water and mixed for two (2) minutes with the L-1 Laboratory Homogenizer. The 100 grams of CWS carbon and 10 grams of pigment were slowly added and blended for five (5) minutes. The slurry is vacuum filtered and the cake dried for four (4) hours at 220-250°F. The resulting filter cake was broken up using a mortar and pestle and screened through a 40 mesh sieve to give the desired consistency.

In preparing the dry formulations the following general procedure was used.

Formulation:            5 grams of 4A3 asbestos  
                             100 grams of CWS carbon  
                             10 grams of iron oxide pigment

### Operations:

The 4A3 asbestos was milled the same as for the wet mixes. The asbestos carbon and pigment were then hand mixed with a spatula to distribute the small amount of asbestos throughout the carbon. The mixture was then put through the pulverizer. Some problem was experienced with the separation of the pigment from the mixture by the pulverizer. The dry mixes appeared to contain considerably more carbon dust or "fly" and did not visually appear to be as homogeneous as did the wet mixes. When screened through a 40 mesh sieve, small amounts of asbestos remained on the screen, indicating the

pulverizer did not give sufficient mixing or dispensing of the asbestos throughout the formulation.

To check the uniformity of the mixer, samples were analyzed for variation in particle size using the Sub-Sieve Analyzer. Results show the wet mixes to be very uniform in that particle size did not vary more than .5 microns in the three samples tested, while the dry mixes varied from 1.5 to 10 microns. This would indicate that clusters of asbestos are present.

As a further check to compare the wet and dry mixes, samples of each of the formulations prepared were placed in clean, dry glass containers for dust and adherence tests.

NOTE: This test, suggested by Edgewood Arsenal, is designed to test the amount of dusting (or fly) present in a carbon/asbestos mix, plus any adherence of the mixture to the clean, dry glass wall of the container would indicate a high concentration of the asbestos. After agitation the mixture should be completely settled with 60 seconds with little or no material adhering to the glass walls.

Results of the glass container tests again indicated the wet formulation to be more uniform than were the dry mixes.

### 3. Abrasion Tests

In order to test the adherence of the carbon/asbestos mixture to woven fabric such as uniform material, a test apparatus was constructed according to the design suggested by Edgewood Arsenal. Figure 21 shows the test apparatus (A) which is designed to tumble the sample holder (B) to which fabric test strips have been attached. Figure 22 is a close-up of the foam cover test cylinder with the fabric samples attached.

The abrasion testing was divided into four basic areas of investigation.

- (1) The effects of wet and dry formulations on the percent carbon retention.
- (2) The effect of formulation moisture content on the percent carbon retention.
- (3) The effect of preparing the asbestos using different screens on the percent carbon retention.
- (4) The percent carbon retention to 6 oz. poplin, 8.2 oz. sateen cotton, and 9 oz. Quarpel-treated Nyco sateen uniform materials.





FIGURE 21  
ABRASION TEST APPARATUS

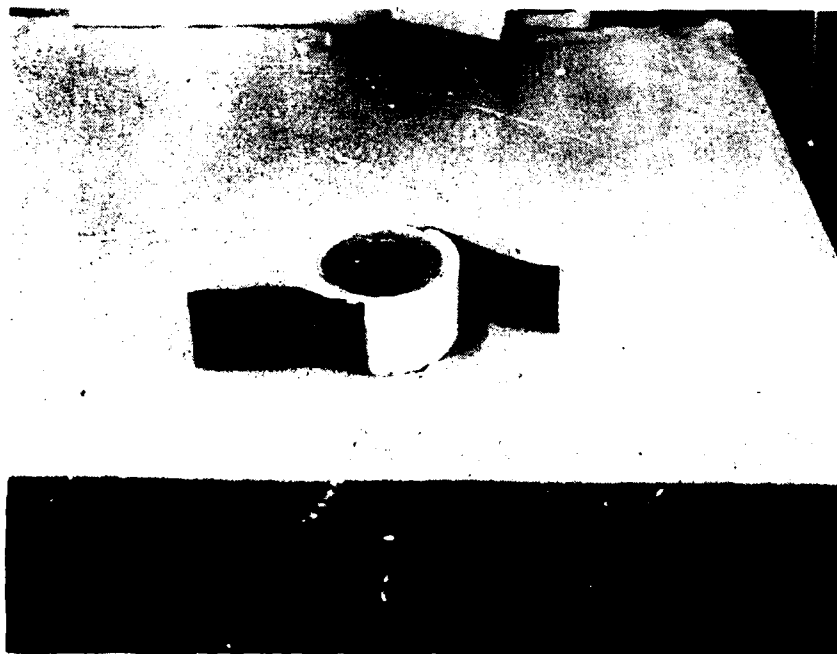


FIGURE 22  
FOAM COVERED TEST CYLINDER  
WITH FABRIC SAMPLES

(a) Test Conditions

Cloth strips were tailored to 5.08 x 10.16 cm and impregnated with CWS carbon/asbestos by using a nylon scrim/cotton bag.<sup>2</sup> An average of 8-12 rubs were required to attain the 4 mg/cm<sup>2</sup> coverage. An after-rub with a clean paper tissue was used to remove excess mixture.

As suggested by Edgewood Arsenal, testing was conducted at 20 RPM. However, as can be seen in Test 1, 2, and 3, shown in Table XXXVIII, retention of carbon mixture on the cloth strips after 30 seconds at 20 RPM exceeded 90%. It was concluded from these data that this test was not severe enough to effectively evaluate the four basic areas previously outlined. In order to compare data collected in earlier published work by Edgewood, the conditions were increased to a more severe 65 RPM for 120 seconds.

Tests 4, 5, and 6 were conducted under these conditions with results also shown in Table XXXVIII.

It was then decided to conduct the remaining screening tests at 65 RPM and 120 seconds.

TABLE XXXVIII

PERCENT CARBON RETENTION VS. RPM OF TEST APPARATUS

<u>Test No.</u>	<u>RPM</u>	<u>Test Time, Sec.</u>	<u>Initial Coverage, mg/cm<sup>2</sup></u>	<u>Percent Retention</u>
1	20	30	4.7	94.3
2	20	30	3.8	92.4
3	20	30	4.7	90.2
4	65	120	3.9	66.2
5	65	120	3.8	69.0
6	65	120	4.3	66.7

NOTES:

- Each of the above values is an average of two (2) samples.
- Tests were performed on 6 oz. poplin material.

(b) Effects of Wet and Dry Prepared Formulations

Abrasion tests were conducted on formulations as previously described, with the more severe test conditions of 65 RPM for 120 seconds.

As can be seen by the results shown in Table XXXIX, the formulations prepared in the aqueous medium gave the higher retention percentages. This again points up the fact that wet formulations are more uniformly mixed than are the dry formulations.

TABLE XXXIX

PERCENT CARBON RETENTION OF WET AND DRY PREPARED FORMULATIONS AT 65 RPM AND 120 SECONDS ABRASION

<u>Test No.</u>	<u>Initial Coverage</u>	<u>Formulating Condition</u>	<u>Percent Retention</u>
4	3.9	Wet	66.2
5	3.8	Wet	69.0
6	4.3	Wet	66.7
7	4.1	Dry	61.8
8	4.4	Dry	59.6
9	4.2	Dry	59.5

(c) Effect of Moisture Content

To measure any effect of moisture content of a carbon/asbestos mixture on the percent retention to woven fabric, mixtures were prepared using the wet method. After preparing the formulation a portion was allowed to reach equilibrium at 72°F and 50% R.H. After twelve hours the moisture content measured 1.8%. After 24, 48, and 96 hours the moisture content had stabilized at 2.0%. The moisture content of the CWS carbon, as supplied by Edgewood, is approximately 2.0%.

To obtain the 0.5% moisture, a sample was dried for four (4) hours at 220-250°F. The remaining moisture contents were obtained by adding calculated amounts of water to reach each desired moisture content and allowing approximately 24 hours to reach equilibrium.

Abrasion tests were ten conducted at 65 RPM and 120 seconds as shown in Table XL.

By evaluating this data, it can be concluded that no significant increase in carbon retention results from increasing the percent moisture. The data also shows that approximately 2% water, which is the normal condition of the carbon as received, exhibits good or slightly superior retention.

TABLE XL

EFFECT OF MOISTURE CONTENT OF A CRABON/ASBESTOS MIXTURE  
ON THE PERCENT RETENTION TO WOVEN FABRIC

65 RPM and 120 Seconds

<u>Test No.</u>	<u>Initial Coverage</u>	<u>Moisture Content</u>	<u>Percent Retention</u>
5	3.8	.5	67.3
6	4.3	2.0	69.0
12	4.5	7.8	59.2
13	4.4	8.0	66.9
14	4.0	7.8	68.0
15	4.0	12.5	63.5

(d) Effect of Pulverizing Screen Size

As discussed earlier, samples of crysotile were pulverized through a series of fine screens. To test the effects of different lengths of asbestos fibers on the percent retention to woven fabric, abrasion tests were conducted as shown in Table XLI. In reviewing the data it can be concluded that there was relatively little effect of the fiber length on the retention of the mixture. Fibers ranging from a few microns to several thousand microns are capable of entangling with each other and entrapping carbon particles.

Based on the microscopic examination showing uniformity of the fiber lengths produced by the .032" round screen, it was selected to produce asbestos for the remaining tests.

TABLE XLI  
EFFECTS OF PULVERIZING SCREEN SIZE ON PERCENT  
CARBON RETENTION TO WOVEN FABRIC

<u>Test No.</u>	<u>Initial Coverage</u>	<u>Screen Size</u>	<u>Percent Retention</u>
16	4.4	.010" x .500" (S)*	64.2
17	4.5	.010" x .500" (S)*	66.3
18	4.5	.010" x .500" (S)*	70.0
4	3.9	.032" (R)*	66.2
5	3.8	.032" (R)*	69.0
6	4.3	.032" (R)*	66.7
19	4.3	.040" (R)*	69.7
20	4.5	.040" (R)*	63.3
21	4.3	.040" (R)*	69.9

NOTE: Formulation prepared as described in Section 2.  
Abrasion Tests run at 65 RPM and 120 seconds.  
\*(S) = Slotted; (R) = Round

(e) Effects of Different Types of Woven Fabric

To determine the percent carbon retention on each of the poplin, 6 oz.; cotton sateen, 8.2 oz.; and Quarpel-treated Nyco sateen, 9 oz., uniform fabrics, abrasion tests were conducted and results shown in Table XLII. As tests 24 and 25 show, the Quarpel-treated Nyco sateen retained 77% to 79% after being abraded for 120 seconds at 65 RPM. This is considerably higher than either the 6 oz. poplin or the 8.2 oz. cotton sateen and probably be explained as the difference in weave of the fabric. The Nyco sateen is a much heavier, more coarse fabric which allows more carbon to penetrate into the voids between the individual strands.

Figures 23, 24, and 25 show Quarpel-treated Nyco sateen as received, impregnated with a carbon/asbestos, and after abrasion testing. As can be seen in Figure 24, the mixture covers the entire surface of the fabric at a level of 4 mg/cm<sup>2</sup>. Figure 25 shows the results of abrading for 120



FIGURE 23

QUARPEL-TREATED NYCO SATEEN AS RECEIVED

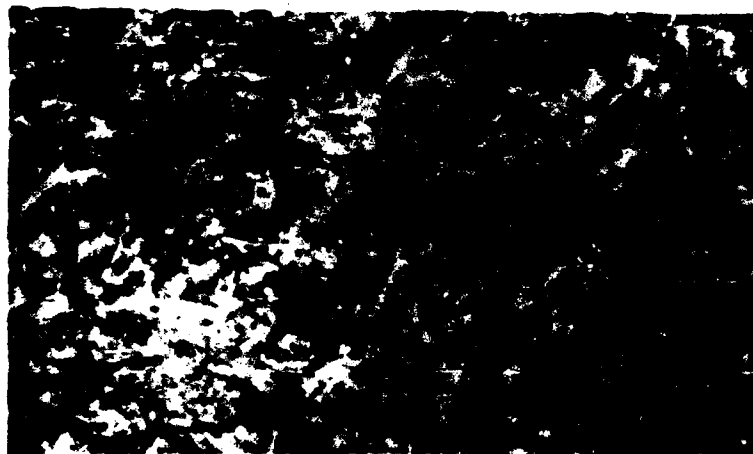


FIGURE 24

NYCO SATEEN IMPREGNATED WITH 4 MG/CM<sup>2</sup>  
OF CARBON/ASBESTOS MIXTURE

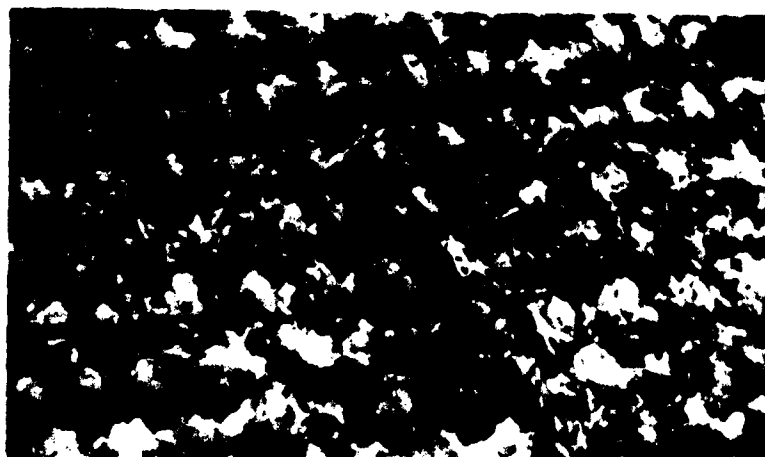


FIGURE 25

NYCO SATEEN AFTER ABRASION TEST

seconds at 65 RPM. Most of the surface carbon has been removed with the remaining portion being entrapped by a combination of the asbestos fibers and weave voids.

TABLE XLII  
PERCENT RETENTION OF CARBON/ASBESTOS  
TO DIFFERENT TYPES OF WOVEN UNIFORM FABRIC

<u>Test No.</u>	<u>Initial Coverage</u>	<u>Type Fabric</u>	<u>Percent Retention</u>
5	3.8	Poplin. 6 oz.	69.0
6	4.3	Poplin, 6 oz.	66.7
22	4.3	Sateen, 8.2 oz.	58.7
23	4.5	Sateen, 8.2 oz.	61.8
24	4.2	Nyco Sateen, 9 oz.*	77.3
25	4.5	Nyco Sateen, 9 oz.*	78.0

NOTES: Formulation prepared as described in Section 2.

Abrasion test run at 65 RPM and 120 seconds.

\*Quarrel-treated Nyco sateen, 9.0 oz.

#### 4. Other Fibrous Fillers

Preliminary evaluation of fibrous materials such as nylon, milled glass and polyethylene was completed. Typical formulation began with 85-90% active carbon, 5-10% pigment and 5% minimum filler. Formulating was conducted using the wet mixing technique as described in Section B-3 of this report.

Samples of the above formulations were submitted to Edgewood Arsenal for evaluation and testing. It was concluded that sufficient work had been completed with fillers to eliminate all but asbestos. At the direction of Edgewood, materials evaluation was limited to asbestos and coal based activated carbon.

### C. Controlling Carbon Flow From Applicator Bags

In order to deposit the desired  $4 \text{ mg/cm}^2$  of active carbon onto a surface, using an applicator bag, it would appear desirous to incorporate some type of flow control device. It was considered that by incorporating large open cell polyurethane foam inside the pad the resulting carbon flow could be better controlled to the desired deposition. However, three problem areas appear to prohibit this concept. First, no satisfactory method could be found whereby 120 grams of the carbon/asbestos could be impregnated into the polyurethane foam. Second, by including the foam into the applicator pad, the volume is increased to the point that it will not fit easily into the M-13 container, and third, the polyurethane foam has a tendency to "filter" or separate the asbestos fibers from the activated carbon. It is felt that by adjusting the nylon "window" scrim, the carbon flow during application can be properly controlled. The optimum size, at this point, appears to be  $2\frac{1}{2}'' \times 4''$ . This allows a  $4 \text{ mg/cm}^2$  coverage level to be attained with minimum rubbing.

### IV. CONCLUSIONS AND RECOMMENDATIONS

Although the water soluble carboxy vinyl polymer thixotropes do provide a stable non-settling, sprayable carbon slurry, gas agent tests have shown that film forming properties adversely effect the important absorptive properties of the activated carbon. Thus, the vinyl polymer system is not suitable for application as a chemical agent decontaminant.

In selecting the optimum ingredients to prepare a stable, sprayable thixotrope that meets all the requirements of the Edgewood specification, the following materials have been shown to be superior.

Coal-based, petroleum-based, coconut shell-based and wood-based carbon all appear to be suitable as gas absorbent decontaminants; however, in considering stability at static storage, the coal-based or the petroleum-based appear to be superior.

Thixotropic additives appear to be limited to the inorganic type with the bentonite showing superior properties to any tested.

Dispersants are effective in lowering the viscosity of a carbon loaded thixotrope, thus allowing the maximum percent carbon loading to retain sprayability. Results show the polymerized salt of alkyl naphthalene sulfonic acid to be best when combined with the coal-based carbon and sodium salts of polymerized substituted benzoid alkyl sulfonic acid to be most effective with the remaining three activated carbon.



Yellow iron oxide pigment, loading to approximately 5% by weight, gives an olive green color when sprayed onto a surface and allowed to dry.

Corrosion and rust can be inhibited by 1/2% by weight of sodium chromate or sodium silicate. Sodium silicate does add to the stability of the petroleum based carbon and is recommended over the sodium chromate with that particular carbon.

A mixture of 40 parts by volume methyl alcohol and 60 parts by volume demineralized water is an effective anti-freeze/carrier system.

The formulation ingredients and composition weights for each of the different based carbons, along with physical characteristics of each, are shown in the formulation and experiments in Section II-C.

In preparing dry powder formulations, Serpentine type asbestos is superior to the Amphibole type in promoting adhesion to woven fabrics.

Formulations prepared in aqueous medium show a higher degree of uniformity than those prepared by simply mixing dry.

Preparation of the asbestos filler can be satisfactorily done by use of a hammer mill fitted with a fine (.032" round holes) screen.

Abrasion tests show active carbon retention to woven fabric to be in the 70% range after a simulated wear of 24 hours.

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## DOCUMENT CONTROL DATA - R &amp; D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Brunswick Corporation Technical Products Division Marion, Virginia 24354		2a. REPORT SECURITY CLASSIFICATION Unclassified	
3. REPORT TITLE Compounding Techniques for Absorbent Decontaminants		2b. GROUP N/A	
4. DESCRIPTIVE NOTES (Type of report and inclusive dates) Final Report (August 1970 - November 1971)			
5. AUTHOR(S) (First name, middle initial, last name) Dewey P. Parks and Robert L. Copeland			
6. REPORT DATE January 1972		7a. TOTAL NO. OF PAGES	
6a. CONTRACT OR GRANT NO. DAAA15-70-C-0362 ✓		7b. NO. OF REFS	
6b. PROJECT NO.		8a. ORIGINATOR'S REPORT NUMBER(S) EACR 1310-3 BR-303	
6c.		8b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report) None	
10. DISTRIBUTION STATEMENT Distribution limited to U.S. Government agencies only because it covers the test and evaluation of military hardware (Jan. 1972). Other requests for this document must be referred to Commanding Officer, Edgewood Arsenal, Attention: SMJEA-TS-R, Edgewood Arsenal, Maryland 21010.			
11. SUPPLEMENTARY NOTES Chemical Physical Protection Investigation		12. SPONSORING MILITARY ACTIVITY Development & Engineering Directorate Edgewood Arsenal Edgewood Arsenal, Maryland 21010 Contract Proj. Officer, Mr. Marshall F. Gilchrist Ext. 2965	
13. ABSTRACT The objective of the program was to develop decontaminating systems for persistent chemical agents using micronized carbon. These systems must be effective in absorbing chemical agents and designed to the following goals: (1) <ul style="list-style-type: none"> <li>Act rapidly over a wide temperature range, (2)</li> <li>Be non-toxic and remain stable, (3)</li> <li>Not damage material and equipment on contact, and (4)</li> <li>Be easily and rapidly applied to vehicular equipment, clothing and ancillary equipment.</li> </ul> The method of study was to select key ingredients, i.e., activated carbons, thixotropic agents, dispersants, pigments and solvent/antifreeze and formulate a system containing a minimum of 25% active carbon. It should be easily applied by spraying to a coverage of 4 m <sup>2</sup> /cm <sup>2</sup> and remain stable (not settle during static storage of at least two years). Key methods of investigation included evaluation of mixing techniques, effects of various thixotropes and dispersant on viscosities and stability and the evaluation of spray equipment for optimization of techniques as well as equipment. In addition, studies were conducted whereby dry formulation of the same key ingredients were formulated using high shear dry mixers. These compounds were tested for uniformity, adhesion to woven surfaces and effective coverages in the			

DD FORM 1473  
1 NOV 65

-107-

Unclassified

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next page

14.	KEY WORDS	LINK A		LINK B		LINK C	
		ROLE	WT	ROLE	WT	ROLE	WT
	<p>4 mg/cm<sup>2</sup> range.</p> <p>The results of these studies show that formulations containing approximately 35% active carbon can be easily sprayed to the desired coverage and will remain stable for the desired two year period. All of the program goals outlined above were met or exceeded by this study. Results also show dry formulations can be prepared by a special screened milling process to give the desired uniformity, color and adhesion.</p> <p>It is concluded that in selecting the optimum ingredients to prepare a stable, sprayable thixotrope that will meet all of the Edgewood requirements, the following materials have shown to be superior: coal, petroleum, coconut and wood based activated carbons are suitable as agent absorbents; however, the coal and petroleum-based appear slightly superior. Thixotropes appear to be limited to the inorganic type with bentonite showing superior thixotropic properties. Dispersants are effective in lowering viscosities with the sodium salts of polymerized substituted benzoid alkyl sulfonic acid being most effective. Color can be obtained with iron oxide and 1/2% sodium chromate or sodium silicate to inhibit rust. A homogenizer mixer will produce uniform mixtures of the wet-system and a special screen milling machine will prepare satisfactory dry formulations.</p> <p>14. Key Words</p> <ul style="list-style-type: none"> <li>Activated Carbon</li> <li>Decontaminants</li> <li>Asbestos</li> <li>Adherence</li> <li>Filler</li> <li>Homogenizer</li> <li>Pulverizer</li> <li>Woven Fabric</li> <li>Pigment</li> <li>Thixotropes</li> <li>Dispersants</li> <li>Corrosion Inhibitors</li> <li>Viscosities</li> <li>Slurries</li> </ul>						